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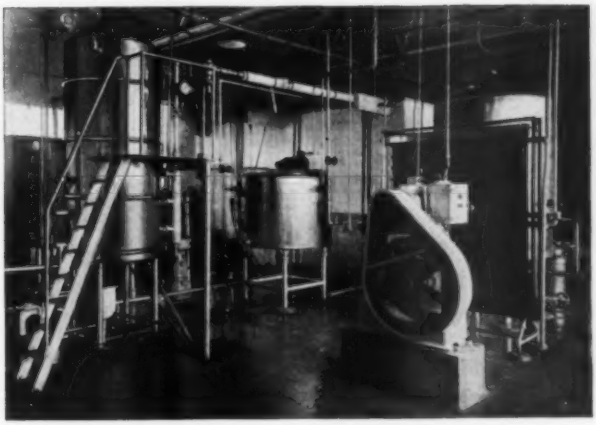

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JOURNAL OF DAIRY SCIENCE

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A STUDY OF METHODS FOR THE DETERMINATION OF ACIDITY IN BUTTER FAT*

D. F. BREAZEALE AND E. W. BIRD

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During the past three years, a study of butter made from neutralized cream has received considerable attention in this laboratory. In connection with this project it seemed possible that valuable information might be obtained by determining the free acidity in the fat phase of the butter. The method employed is based on the general method given by Leathes (3). In addition, the details of the Leathes method for the quantitative separation of free acids from butter fat have been worked out because it is desirable at times to obtain these acids free from fat for further study. The following experiments were undertaken in order to check these methods quantitatively and to compare them with the Official method of the A. O. A. C. (1) and the recently described method of Clarke *et al.* (2).

EXPERIMENTAL

Reagents and apparatus. Petroleum ether was distilled over solid KOH and the fraction which boiled between 30° and 40° C. was used. Benzene was likewise distilled from solid KOH. Absolute alcohol was refluxed for 1 to 2 hours over solid KOH and aluminum metal (10 gm. of each per liter) and then distilled from these reagents (6). Alcohol so purified was used in the preparation of 0.5 N KOH and C_2H_5ONa solutions. Indicator solutions were prepared as follows: 1 gm. of phenolphthalein was dissolved in 100 ml. of absolute alcohol after which alcoholic KOH was added until a faint pink color was produced. Ordinary 95 per cent alcohol was made very slightly alkaline to phenolphthalein with 0.1 N NaOH for the A. O. A. C. method.

Each of the alkaline solutions was protected from CO_2 to minimize changes in concentration. A siphon from the reservoir was connected to a side arm at the base of the burette; the top of the burette was connected to a T-tube which extended just through the stopper into the reservoir, while a soda-lime tube was joined to the other arm of the T-tube. The alkaline solutions were

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standardized against 0.01 N HCl at frequent intervals; the greatest change in normality in a three month period was 0.0003. The HCl solution was standardized gravimetrically by precipitating and weighing the chlorine as AgCl.

All samples of fat for titration were weighed on a Torsion butter moisture balance. The sensitivity of this balance was such that 0.02 gm. displaced the zero reading 0.5 scale space. It is therefore considered that the samples could be weighed to $\pm$ 0.01 gm. of the desired weight. In all cases this would represent an amount of acid too small to be of any significance in the titrations.

The acids and fat (in the preparation of fat-acid mixtures) and the extracted acids were weighed on an Ainsworth type TC balance. The weighings were estimated to 0.01 mg. by the method of swings.

An International size 2 centrifuge was used in the separation of acids from fat.

Methods. The acidity determinations were made on two types of materials. In one type, the acids were removed as completely as possible from a large sample (about 1000 gm.) of high quality butter fat and definite mixtures by weight of this fat and various fatty acids were prepared; in the second, the filtered fats from samples of market butter secured from several sources were employed.

The acid-free fat was prepared as follows: The butter was heated in a water bath at 55° to 60° C. until a clear fat sample was obtained when filtered. Lots of about 130 gm. of this fat were weighed into 150 ml. beakers, transferred to 1 liter separatory funnels with 400 ml. of petroleum ether. Then 160 ml. of absolute alcohol and 10 drops of phenolphthalein were added and the acids were neutralized with 1 N alcoholic KOH. In order to minimize hydrolysis of the soaps formed, 10 ml. of NH_4OH (28 per cent NH_3) were added and mixed with the solution; 150 ml. of H_2O were added next and the contents were mixed by rotating for 2 minutes. When separation appeared to be complete, the lower layer containing the soaps was removed. One hundred milliliters of absolute alcohol were added to and mixed with the fat-ether solution, after which 100 ml. of H_2O were added, the funnels were shaken vigorously for 2 minutes and the aqueous-alcoholic layer was again removed. This process was repeated using 50 ml. each of absolute alcohol and water and was followed by two washings with 100 ml. of water alone. After the water was removed as completely as possible, the fat solutions were poured from the separatory funnels into large Erlenmeyer flasks and were dried for several hours with anhydrous Na_2SO_4. The solution was filtered and the petroleum ether was distilled from the fat; the last traces of petroleum ether were removed by heating in the oven of the Mojonnier tester at 100° C. and 20 in. vacuum for 30 minutes.

The saturated acids employed in these experiments were obtained from

Eastman Kodak Company and were used without further purification. The oleic acid was partially purified by crystallization of the lithium salt from 50 per cent alcohol (5) and was then distilled under reduced pressure. However, it is evident from the neutralization equivalent obtained that it was not pure.

The insoluble butter acids were prepared by boiling 10 gm. of butter fat and 75 ml. 1 N alcoholic KOH under reflux for 30 minutes, removing the reflux condenser, adding 200 ml. of H_2O and continuing the heating until the alcohol had been removed. The soap solution was diluted to about 800 ml. with boiling water and was acidified with dilute H_2SO_4. This mixture was heated until the acids separated as an oily layer on the surface, after which the beaker and its contents were cooled until the solid cake of insoluble acids could be transferred to another beaker of boiling water. The alternate heating and solidification process was repeated until there was very little odor of the lower molecular weight acids. Moisture was removed from the solid cake of acids by pressing between layers of filter paper. The acids were next dissolved in petroleum ether and the excess water was removed in a separatory funnel. The solution was dried for several hours over Na_2SO_4, filtered, the bulk of the petroleum ether was evaporated and the last traces of it were removed in the vacuum oven at 100° C. and 20 in. vacuum for 30 minutes.

The methods employed to determine the amounts of free acids in the fat-acid mixtures and in the ordinary samples of butter fat are:

A. Official method of the A. O. A. C. (1). Twenty grams of fat were weighed into 125 ml. Erlenmeyer flasks, 50 ml. of neutralized alcohol and 10 drops of phenolphthalein were added, the contents were heated to boiling, the flasks were shaken vigorously and the titrations were made with 0.1 N NaOH. An attempt was made to obtain an end point which would persist for 30 seconds.

B. Method of Clarke *et al.* (2). Five grams of fat were weighed into 125 ml. Erlenmeyer flasks, 15 ml. of benzene and 5 drops of phenolphthalein were added and the titrations were made with 0.05 N C_2H_5ONa in absolute alcohol. Blank titrations on the benzene were subtracted from the burette readings of the samples.

C. Alcoholic KOH method (A). Ten grams of fat were weighed into 125 ml. Erlenmeyer flasks and dissolved in 25 ml. of petroleum ether; 10 ml. of absolute alcohol and 10 drops of phenolphthalein were added and the titrations were made with 0.05 N KOH in absolute alcohol. The first definite color change (compared with a sample which had not been titrated) was taken as the end-point. Blank determinations on the reagents were subtracted from the burette readings of the samples.

D. Alcoholic KOH method (B). Ten grams of fat were weighed into 50 ml. beakers, transferred to 150 ml. Squibb type separatory funnels (designed for use in the centrifuge) with 50 ml. of petroleum ether; 25 ml. of

absolute alcohol and 10 drops of phenolphthalein were added and the titrations were made as described in C. One milliliter of NH_4OH (28 per cent NH_3) was added and mixed with the solution. An amount of water equal to the total amount of alcohol present was added, the funnels were stoppered, shaken vigorously for 2 minutes and were centrifuged at approximately 1000 revolutions per minute until the emulsion had been broken—usually 5 minutes was sufficient. The lower layers (solutions of soaps) were run into 250 ml. centrifuge bottles. Ten milliliters of absolute alcohol were added to and mixed with the fat solution, 10 ml. of H_2O were added and the funnels were again shaken vigorously and centrifuged. The washing of the fat was repeated using 5 ml. each of absolute alcohol and water. The combined soap solution and washings were twice shaken vigorously for about 2 minutes with 50 ml. portions of petroleum ether and were centrifuged at 1500 to 1800 revolutions per minute for 15 to 20 minutes to free them from fat. The petroleum ether layer was forced from the bottle through a small glass tube (fitted in a two-hole rubber stopper) by means of air pressure from an atomizer bulb. The petroleum ether was blown into a small separatory funnel and the small amounts of soap solution frequently included were returned to the centrifuge bottles. The soap solutions were acidified with 3 ml. of H_2SO_4 (1 part H_2SO_4 to 4 parts H_2O by volume). The solutions at this stage should be strongly acid to methyl orange. The free fatty acids were extracted as follows: The acidified solutions were shaken with three successive portions (50, 25, and 25 ml. respectively) of petroleum ether; after each treatment the flasks were centrifuged and the petroleum ether layer was drawn off. The combined extracts were washed with 50 ml. H_2O in 250 ml. separatory funnels to remove traces of H_2SO_4. These acid extracts were poured into weighed 125 ml. Erlenmeyer flasks, the petroleum ether was slowly evaporated on an electric hot plate under the hood and the last traces were removed in the vacuum oven at 85° to 90° C. and 20 in. vacuum for 15 to 20 minutes. After cooling the flasks were reweighed; the recovered acids were dissolved in 50 ml. of petroleum ether, 25 ml. of absolute alcohol and 10 drops of phenolphthalein were added and the titrations were made with 0.05 N alcoholic KOH solution. Blank determinations made on the solvents were subtracted from the samples.

The neutralization equivalents of the fatty acids used in these experiments were determined by the method of Sandin, Kulka and Woollney (4). The C_2H_5ONa solution was standardized against 0.01 N HCl rather than highly purified stearic acid as recommended by these authors. The calculated titration values were then obtained as follows:

$$\text{Calculated ml. 0.1 N alkali} = \frac{\text{Weight of acid} \times 10{,}000}{\text{Neutralization equivalent}}$$

Results. The analytical data obtained with the above methods are presented in Tables 1 and 2. The two alcoholic KOH methods, whose values are nearly identical, agree more closely with the calculated values than do

TABLE 1

Acidity determinations of butterfat—fatty acid mixtures

Kind of acid added		1 Neut. equiv. of acids added to fat	Titration values: ml. 0.1 N alkali per 10 gm. sample						8 Weight of acid added per 10 gm. sample	9 Weight of acid recovered	10 % of weight of added acid recovered	11 Titrat. recovered acids as % orig. titrat. $\frac{\text{col. 7}}{\text{col. 6}} \times 100$
			2 Calc. from neut. equiv. and weight of acid	3 A.O.A.C. method	4 Method of Clarke *et al.*	5 Alcoholic KOH method[1] (A)	6 Alcoholic KOH method[2] (B)	7 Recovered acids				
None	a			0.10	0.02	0.05	0.05	0.01		0.00102		
	b			0.10	0.02	0.04	0.04	0.01		0.00112		
	av.			0.100	0.020	0.045	0.045	0.010		0.00107		22.2
Lauric	a			2.36	2.45	2.35	2.33	2.22		0.04615		
	b			2.38	2.42	2.34	2.34	2.25		0.04656		
	av.			2.370	2.435	2.345	2.335	2.235		0.04636		
	corr.[3]	201.2	2.290	2.270	2.415	2.300	2.290	2.225	0.04607	0.04529	98.3	97.2
Myristic	a			2.25	2.34	2.24	2.23	2.16		0.05030		
	b			2.27	2.33	2.24	2.24	2.17		0.05019		
	av.			2.260	2.335	2.240	2.235	2.165		0.05025		
	corr.[3]	225.8	2.218	2.160	2.315	2.195	2.190	2.155	0.05009	0.04918	98.2	98.4
Palmitic	a			1.96	2.04	1.94	1.95	1.87		0.04929		
	b			1.96	2.06	1.94	1.94	1.87		0.04937		
	av.			1.960	2.050	1.940	1.945	1.870		0.04933		
	corr.[3]	255.7	1.910	1.860	2.030	1.895	1.900	1.860	0.04883	0.04826	98.8	97.9
Stearic	a			1.86	1.91	1.83	1.82	1.74		0.05103		
	b			1.85	1.90	1.83	1.84	1.75		0.05146		
	av.			1.855	1.905	1.830	1.830	1.745		0.05125		
	corr.[3]	282.7	1.790	1.755	1.885	1.785	1.785	1.735	0.05049	0.05018	99.4	97.2
Oleic	a			1.90	1.94	1.86	1.85	1.76		0.05114		
	b			1.89	1.93	1.86	1.85	1.76		0.05129		
	av.			1.895	1.935	1.860	1.850	1.760		0.05122		
	corr.[3]	284.9	1.815	1.795	1.915	1.815	1.805	1.750	0.05172	0.05015	97.0	97.0

TABLE 1—(*Continued*)

Kind of acid added		1 Neut. equiv. of acids added to fat	Titration values: ml. 0.1 N alkali per 10 gm. sample						8 Weight of acid added per 10 gm. sample	9 Weight of acid recovered	10 % of weight of added acid recovered	11 Titrat. recovered acids as % orig. titrat. col. 7 / col. 6 × 100
			2 Calc. from neut. equiv. and weight of acid	3 A.O.A.C. method	4 Method of Clarke *et al.*	5 Alcoholic KOH method[1] (A)	6 Alcoholic KOH method[2] (B)	7 Recovered acids				
Insoluble butter acids	a			0.51	0.50	0.45	0.45	0.38		0.01130		
	b			0.52	0.50	0.46	0.45	0.38		0.01117		
	av.			0.515	0.500	0.455	0.450	0.380		0.01124		
	corr.[3]	268.7	0.400	0.415	0.480	0.410	0.405	0.370	0.01075	0.01017	94.6	91.4
Insoluble butter acids	a			1.96	1.96	1.87	1.87	1.76		0.04836		
	b			1.96	1.95	1.87	1.87	1.76		0.04817		
	av.			1.960	1.955	1.870	1.870	1.760		0.04827		
	corr.[3]	268.7	1.827	1.860	1.935	1.825	1.825	1.750	0.04909	0.04720	96.2	95.9
Insoluble butter acids	a			3.86	3.90	3.79	3.80	3.63		0.09894		
	b			3.84	3.91	3.80	3.80	3.60		0.09839		
	av.			3.850	3.905	3.795	3.800	3.615		0.09867		
	corr.[3]	268.7	3.764	3.750	3.885	3.750	3.755	3.605	0.10115	0.09760	96.5	96.0

[1] 10 gm. sample weighed into 125 ml. Erlenmeyer flask, dissolved in 25 ml. petroleum ether, 10 ml. absolute alcohol added and titrated with KOH in absolute alcohol.

[2] 10 gm. sample weighed into 50 ml. beaker, transferred to 150 ml. separatory funnel with 50 ml. petroleum ether, 25 ml. absolute alcohol added and titrated with KOH in absolute alcohol.

[3] The average value of the sample containing no added acid was subtracted from the averages of the other samples to obtain the corrected values.

TABLE 2

Acidity determinations of butterfat from samples of commercial butter

Sample number		Titration values: ml. 0.1 N alkali per 10 gm. fat					6 Weight of recovered acids	7 Titrat. recovered acids as % orig. titrat. $\frac{\text{col. 5}}{\text{col. 4}} \times 100$
		1 A.O.A.C.	2 Method of Clarke *et al.*	3 Alcoholic KOH method[1] (A)	4 Alcoholic KOH method[2] (B)	5 Recovered acids		
	a	0.50	0.59	0.56	0.56	0.51	0.01392	
1	b	0.51	0.60	0.56	0.55	0.52	0.01375	
	av.	0.505	0.595	0.560	0.555	0.515	0.01384	92.8
	a	0.75	0.78	0.72	0.73	0.69	0.01863	
2	b	0.76	0.78	0.73	0.73	0.69	0.01859	
	av.	0.755	0.780	0.725	0.730	0.690	0.01862	94.5
	a	1.34	1.42	1.34	1.36	1.28	0.03473	
3	b	1.34	1.43	1.34	1.34	1.28	0.03470	
	av.	1.340	1.425	1.340	1.350	1.280	0.03472	94.8
	a	0.71	0.78	0.72	0.71	0.67	0.01814	
4	b	0.71	0.78	0.71	0.72	0.67	0.01817	
	av.	0.710	0.780	0.715	0.715	0.670	0.01816	93.7
	a	0.74	0.80	0.74	0.76	0.72	0.01916	
5	b	0.73	0.80	0.74	0.74	0.72	0.01936	
	av.	0.735	0.800	0.740	0.750	0.720	0.01926	96.0
	a	0.74	0.79	0.72	0.74	0.67	0.01805	
6	b	0.74	0.78	0.72	0.74	0.67	0.01814	
	av.	0.740	0.785	0.720	0.740	0.670	0.01810	90.5
	a.	0.59	0.66	0.61	0.64	0.57	0.01526	
7	b	0.57	0.68	0.61	0.62	0.56	0.01515	
	av.	0.580	0.670	0.610	0.630	0.565	0.01522	89.7
	a	0.56	0.64	0.59	0.59	0.55	0.01564	
8	b	0.56	0.64	0.59	0.60	0.55	0.01503	
	av.	0.560	0.640	0.590	0.595	0.550	0.01534	92.4
	a	0.70	0.71	0.71	0.67	0.63	0.01720	
9	b	0.75	0.71	0.70	0.68	0.64	0.01710	
	av.	0.725	0.710	0.705	0.675	0.635	0.01715	94.0
	a	0.60	0.65	0.64	0.64	0.58	0.01597	
10	b	0.59	0.65	0.64	0.63	0.58	0.01556	
	av.	0.595	0.650	0.640	0.635	0.580	0.01577	91.3
	a	0.91	0.86	0.81	0.80	0.75	0.02058	
11	b	0.86	0.86	0.80	0.81	0.75	0.02054	
	av.	0.885	0.860	0.805	0.805	0.750	0.02056	93.2
	a	0.63	0.65	0.62	0.62	0.56	0.01566	
12	b	0.70	0.67	0.61	0.62	0.55	0.01524	
	av.	0.665	0.660	0.615	0.620	0.555	0.01545	89.5
	a	0.79	0.78	0.72	0.72	0.66	0.01818	
13	b	0.77	0.76	0.71	0.72	0.66	0.01821	
	av.	0.780	0.770	0.715	0.720	0.660	0.01820	91.6
	a	0.75	0.78	0.75	0.76	0.70	0.01888	
14	b	0.79	0.78	0.75	0.75	0.70	0.01869	
	av.	0.770	0.780	0.750	0.755	0.700	0.01879	92.7
	a	0.47	0.50	0.48	0.50	0.43	0.01222	
15	b	0.44	0.52	0.47	0.50	0.43	0.01192	
	av.	0.455	0.510	0.475	0.500	0.430	0.01207	86.0
	a	1.21	1.33	1.23	1.26	1.14	0.03031	
16	b	1.18	1.34	1.24	1.26	1.14	0.03052	
	av.	1.195	1.335	1.235	1.260	1.140	0.03042	90.5

TABLE 2—(*Continued*)

Sample number		Titration values: ml. 0.1 N alkali per 10 gm. fat					6 Weight of recovered acids	7 Titrat. recovered acids as % orig. titrat. col. 5 / col. 4 × 100
		1 A.O.A.C.	2 Method of Clarke *et al.*	3 Alcoholic KOH method[1] (A)	4 Alcoholic KOH method[2] (B)	5 Recovered acids		
17	a	0.79	0.89	0.84	0.84	0.78	0.02145	
	b	0.84	0.91	0.85	0.84	0.79	0.02160	
	av.	0.815	0.900	0.845	0.840	0.785	0.02153	93.5
18	a	1.65	1.76	1.67	1.67	1.59	0.04288	
	b	1.63	1.76	1.67	1.66	1.60	0.04293	
	av.	1.640	1.760	1.670	1.665	1.595	0.04291	94.8
19	a	1.17	1.27	1.18	1.18	1.10	0.02972	
	b	1.14	1.25	1.18	1.17	1.10	0.02940	
	av.	1.155	1.260	1.180	1.175	1.100	0.02956	93.6
20	a	1.38	1.50	1.40	1.41	1.34	0.03668	
	b	1.38	1.48	1.41	1.42	1.36	0.03691	
	av.	1.380	1.490	1.405	1.415	1.350	0.03680	95.4
21	a	0.65	0.71	0.66	0.67	0.58	0.01635	
	b	0.66	0.70	0.67	0.67	0.57	0.01621	85.8
	av.	0.655	0.705	0.665	0.670	0.575	0.01628	
Averages		0.840	0.898	0.843	0.848	0.787	0.02137	92.1

[1] See table 1.
[2] See table 1.

the methods of Clarke *et al.* and the A. O. A. C. The method of Clarke *et al.* gives somewhat higher values than the calculated while the A. O. A. C. method gives slightly lower values. If these two methods are compared with the alcoholic KOH methods in Table 2, the same relationships hold among the average values. If the average values (uncorrected) from Table 1 are compared, it will be observed that the alcoholic-KOH method still yield values that agree more closely with the calculated values than do those of the other methods. It should be emphasized, however, that the differences between these methods are not very great and probably have little significance—at least for comparative work.

Considerable difficulty was encountered in obtaining good checks between duplicate determinations with the A. O. A. C. method as a result of the rapidly fading end-point. With the method of Clarke *et al.* the end-points were somewhat obscured in the samples containing the pure saturated fatty acids because the soaps formed in the titrations precipitated to a considerable extent. This did not occur appreciably with the other samples in Table 1 or with the samples of ordinary butter fat. It is believed that the alcoholic KOH method (A) offers some advantages over the other two methods in that the end-points persist longer, the color changes are more easily observed, and somewhat better agreement between duplicate determinations is obtained.

No attempt has been made to identify the material extracted in the control sample. Since the method of removing the acids originally present in the

fat so closely resembles the method employed in obtaining the data of Table 1, it is difficult to explain the presence of the acids in the control unless a small amount of hydrolysis of fat occurred after the removal of the original acids. It is possible that this extracted material may contain some fat, phospholipin or both.

The percentages of acids recovered with the extraction method are given in columns 10 and 11 of Table 1 and column 7 of Table 2. The most probable explanations for the 22.2 per cent recovery of the titratable acidity in the control sample are: (a) that more alkali was required to produce a perceptible end-point in the presence of the fat than in a clear solution, and (b) that the fat contained a small amount of volatile acidity which was lost during the extraction process. Table 1 shows that the titratable acidity of the recovered acids (column 7) as percentage of the original titration (column 6) agrees rather well with the percentage of the weight of added acids which was recovered. In Table 2 the titratable acidities of the recovered acids are lower percentage values (column 7) of the original titrations than are the corresponding values of Table 1. No control could be employed here as was done with the values in Table 1; it is logical to assume, therefore, that error resulting from the masking of end-points is largely responsible for the lower values obtained when the titration of the recovered acids is calculated as percentage of the titration in the presence of fat. The samples used for the values of Table 2 were more highly colored than those in Table 1. Volatile acids in the fat may account for a part of the discrepancy.

Some information regarding the purity of the extracted acids can be obtained by calculating their neutralization equivalents from the data given in column 7 and 9 of Table 1 and comparing them with the values given in column 1. These calculations indicate that the acids are at least 97 per cent of their original degree of purity.

SUMMARY AND CONCLUSIONS

1. Methods were described for the titration of free acids in butter fat and for the separation of these acids from the fat. Comparisons of these methods with the A. O. A. C. method and that of Clarke *et al.* were presented for butter fat samples from commercial butters and for samples of fat to which definite weights of fatty acid were added.

2. Good agreement was obtained among the titration methods although the method of Clarke *et al.* gave slightly higher values than the other methods.

3. The titration end-points were most easily observed with the alcoholic KOH methods and the best agreement between duplicates was obtained with these methods. The end-points of the A. O. A. C. method were the most difficult and for this reason the duplicates did not agree as closely as with the other methods.

4. From 94.6 to 99.4 per cent of the weight of added acids were recovered by the acid separation method described. Apparently these acids were at least 97 per cent of their original degree of purity.

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THE EFFECT OF FEEDING MANGELS OR DRIED BEET PULP TO COWS ON THE DEVELOPMENT OF OXIDIZED FLAVOR IN MILK*

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The milk of the Experiment Station Jersey herd has been unusually free from oxidized flavors in comparison with milk from other local sources. This herd, for many years, has been fed mangels during the winter months which is contrary to the usual feeding practice.

It is well known that cows on pasture or cows fed green cut legumes or grasses produce milk which is less susceptible to the development of oxidized flavors. Likewise, it has been shown by Anderson[1] that the feeding of carrots during the winter months tends to prevent the development of this flavor in milk. In view of these results and since feed is one of the many variable factors in the development of this flavor it is logical to assume that the feeding of mangels might be a factor in preventing its development.

Dried beet pulp has never been fed to our herd but is very common in dairy rations. Since Davies[2] reported that the feeding of dried beet pulp sometimes caused a fishy or off-flavor in milk it seemed advisable to ascertain whether or not this flavor might be related to an oxidized flavor.

EXPERIMENTAL PROCEDURE

As soon as the station Jersey herd of 26 milking cows was removed from pasture in the fall weekly samples were taken of approximately one quart of milk from each cow at the noon milking. One pint bottle of each of these samples was pasteurized at a temperature of 143° F. to 145° F. for one-half hour in a water bath. After pasteurization half of each pint of hot milk was poured into half pint bottles containing sufficient copper sulphate solution to increase the copper content by 0.25 p.p.m. It was desirable to add copper to part of the milk to determine more accurately the degree of susceptibility of the milk from each cow to the production of oxidized flavors. The milk was all cooled in the bottles in an ice water bath. The raw milk was judged for flavor by the authors shortly after milking and the raw, pasteurized and pasteurized plus copper milks were judged the first, third, and fifth days after milking and pasteurization.

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[1] The Cause of Rancid and Oxidized Flavors in Raw Milk. J. A. Anderson, Proceedings International Association of Milk Dealers, Lab. Section, pp. 117–134, 1937.

[2] The Fishy Flavor of Milk Caused by Feeding Beet By-products. W. L. Davies, Agricultural Progress 13, 112–5, 1936.

TABLE 1

Average of three weeks' scores of milk when no mangels were fed

Cow No.	Fresh	1 day old		3 days old			5 days old		
	Raw	Past.	Past. plus .25 p.p.m. copper	Raw	Past.	Past. plus .25 p.p.m. copper	Raw	Past.	Past. plus .25 p.p.m. copper
1	23½	22½	22½	22⅝	22½	17½*	22½	20½*	17 *
2	23½	22½	22½	22⅝	22½	17½*	22½	22½	17 *
4	23½	22⅝	22½	23	22½	20 *	22½	22½	19½*
5	23	22⅝	22 *	22⅝	21⅝*	17½*	21⅝	19 *	17½*
6	23½	22⅝	22½	22⅝	22½	20½*	22½	22	20
7	23½	22½	21½†	22	22½	15½*	20½	21½	17½*
8	23½	22⅝	22½	22⅝	22½	16½*	22½	19½	18 *
9	23½	22⅝	22	23	22½	22	22½	22	.....
10	23½	22⅝	22½	22½†	22½	19 *	21 †	21 †	18½*†
11	23½	22⅝	22½	22⅝	22½	18⅝†	22½	22	18½*
12	23½	22⅝	22½	22⅝	22½	19½*	22½	21⅝	18 *
13	23½	22⅝	22½*	22½	22½	18½*	22½	21⅝	18 *
15	23½	22⅝	22½	22⅝	22½	21 *	22½	21⅝	19 *
16	21½†	21 †	20⅝†	21 †	20⅝†	17½*†	20½†	19½†	18½*†
18	23	22⅝	22 *	22	21½	17⅝*	21½†	21 *	20 *
19	23½	22½	21½†	22⅝†	21⅝†	19½*†	21⅝	21½†	18
20	23½	22½	21½*	22⅝	22	16 †	22	19 *	.....
23	23	22½	22	22⅝	22½	21 *	22	22	.....
25	23½	22⅝	22½	22⅝	22½	17½*	22½	22½	19 *
26	23½	22½	22½	22⅝	22½	19½*	22	22½	20½*

* oxidized, † salty. All other cuts for old or heated flavors.

The first three weeks after the cows were off pasture none of the herd received mangels.

RESULTS

The Effect of Feeding Mangels

The milk from each cow was handled and scored for flavor as described in the experimental procedure. Table 1 gives the average score of the milk for three weeks when no mangels were fed. No oxidized flavor developed in any of the samples of raw milk even after five days at 40° F. Only one sample of pasteurized milk developed an oxidized flavor after three days and four samples after five days. The milk from cow number five was most susceptible to the development of oxidized flavors and after the cows had been off pasture for a longer period the raw milk from this cow became oxidized after five days. As shown in table 1 only four of the samples to which copper had been added became oxidized after one day but practically all of them became oxidized on the third and fifth day.

Beginning the following week the cows number 1 to 13, inclusive, were fed mangels. There was no selection in the group and they constitute one row of cows in the barn. The milk from the individual cows and a composite sample of the milk from cows number 1 to 13 and cows 14 to 26 were handled as previously described. The average scores for this three weeks period are given in Table 2. As can be seen from this table the feeding of mangels apparently exerted no influence in preventing the development of oxidized flavors. Likewise the composite samples of the mangel and non-mangel group in Table 2 showed no difference in their susceptibility to the development of oxidized flavors.

Since the first two tables are averages of scores for three week periods they do not give the frequency of occurrence of oxidized flavor for a given number of samples tested. Table 3 gives this information for the two periods of three weeks. In the first three weeks tests the samples containing copper were tasted on the fifth day only during the last week and the first weeks samples did not have copper added to them.

The first half of Table 3 gives the occurrence of oxidized flavor when no mangels were fed. The cows in this table are nevertheless divided into groups 1 to 13 and 14 to 26. The group from 1 to 13 was selected to be fed mangels since the oxidized flavor occurred more often in this group and should mangels help to prevent the development of this flavor it would be more noticeable in this group.

The second part of Table 3 shows the occurrence of oxidized flavors in the same two groups when cows 1 to 13 are fed mangels. The raw milk of cow number 5 developed an oxidized flavor at this time after five days holding and is responsible for the occurrence of this flavor as shown in the table. Table 3 might indicate that the milk from the group receiving mangels was

TABLE 2

Average of three weeks' scores of milk when half the herd received no mangels and half received mangels

Cow No.	Fresh	1 day old		3 days old			5 days old		
	Raw	Past.	Past. plus .25 p.p.m. copper	Raw	Past.	Past. plus .25 p.p.m. copper	Raw	Past.	Past. plus .25 p.p.m. copper
Mangels									
1	23½	23	23	22½	22¾	19¾*	21¾	21¾	15¾*
2	23½	23	23	22½	22¾	16¾*	21½	21½	15½*
4	23	23	23	22½	22½	19 *	21½	21	17½*
5	23¼	23	21¾*	22¼	20¾*	17½*	20¾*	17½*	15 *
6	23¼	23	23	22½	22½	21½*	21½	21½†	18½*
8	22½†	22½†	21 †	21½†	20½*†	17½*†	20½*†	18 *†	16½*
9	23¼	23	23	22½	22¼	21 *	21½	21¼	17¼*
10	23	22¾†	22¾†	22 †	22¼†	20½*	20½†	20¾†	17½*
11	23½	23	23	22½	22¾	17 *	21¾	21¼	15 *
13	23½	23	22¾	22¼	22¾	17 *	21¾	21¾	15½*
1–13	23½	23	23	22¼	22½	18¾*	21½	21¼	17 *
No Mangels									
15	23½	23	23	22½	22¼	21¾	21¾	21¼†	17¾*
16	21½†	21¼	20¾†	20½†	20¼†	17¾*†	19¾†	18¾†	16½*†
18	21 †	21¼†	20½†	20½†	20¼†	20¾*†	20½†	18 †	14¾†
19	23¼	23	23	22½	22¾	18 *	21¾	21¼	16¼*
23	23¼	23	23	22¼	22¾	21¼	21½	21¾	17½*
25	22¾	22¾	21 *	22¼	22¼	17¼*	21¼	21¼†	16 *
26	23½	23	23	22¼	22¾	21¼	21½	21½	17 *
14–26	23½	23	23	22½	22¾	18¾*	21¾	21¾	17¼*

* oxidized, † salty. All other cuts for old or heated flavors.

TABLE 3

Frequency of occurrence of oxidized flavor in milk of individual cows of Station Jersey herd when the herd received no mangels for a period of three weeks followed by half the herd receiving mangels and the other half no mangels for the next three weeks

Age of samples days	Cows 1–13					Cows 14–26				
	Mangel Group					No Mangel Group				
	No. samples tasted	Raw	Past.	No. samples tasted	Past. plus .25 p.p.m. copper	No. samples tasted	Raw	Past.	No. samples tasted	Past. plus .25 p.p.m. copper
1	35	0	0	23	3	24	0	0	14	1
3	35	0	2	23	21	24	0	0	14	10
5	35	0	8	11	11	24	0	2	6	6
	Mangels Fed									
1	30	0	0	30	3	21	0	0	21	2
3	30	0	5	30	23	21	0	1	21	9
5	30	3	6	30	27	21	0	0	21	16

TABLE 4

Average scores for milk samples from Holstein cows that received no dried beet pulp

Cow No.	Fresh	1 day old		3 days old			5 days old		
	Raw	Past.	Past. plus .25 p.p.m. copper	Raw	Past.	Past. plus .25 p.p.m. copper	Raw	Past.	Past. plus .25 p.p.m. copper
27	22½	21½	21½	20½	20½	17 *	20	20	17 *
28	23	23	22½	21½	22	17½*	21½	21½	16½*
29	23	23	18½*	22	19 *	16½*	20½*	16½*	16½*
30	23	23	22½	22	22	21½	21½	21½	20½
31	23	23	22½	22	22	19 *	21½	21½	17½*
32	23	23	22½	22	22	17½*	21½	21½	17 *
33	23	23	21½*	22	22	17 *	21½	21½	16 *
34	23	22½	21½*	21½	21½	17 *	18½	21½	16 *

* oxidized. All other cuts for old or heated flavors.

TABLE 5

Average scores for milk samples from Holstein cows when half of these cows received dried beet pulp

Cow No.	Fresh	1 day old		3 days old			5 days old		
	Raw	Past.	Past. plus .25 p.p.m. copper	Raw	Past.	Past. plus .25 p.p.m. copper	Raw	Past.	Past. plus .25 p.p.m. copper
No Dried Beet Pulp									
27	22¾	22½	21 *	22½	22½	16 *	21¾	22	16 *
28	22¾	22	20½*	22½	22	18½*	21½	21¾	16½*
29	23	22½	21 *	22½	20½*	18 *	18½*	18¾*	17 *
30	23	22½	22½	22½	22	20 *	20	20¾*	17½*
Dried Beet Pulp									
31	23	22½	21 *	22½	21¾	17 *	21¾	20¾*	16½*
32	23	22½	21½*	22¾	22½	20½*	21½	22	17 *
33	23	22½	19½*	20 *	19¾	18 *	21 *	19 *	17½*
34	23	22½	20 *	21½	22½	21	21½	19¾*	16½*

* oxidized. All other cuts for old or heated flavors.

a little more susceptible to the development of the oxidized flavor than the non-mangel group if it were not for the fact that the relationship of the two groups was the same prior to feeding mangels.

These experiments indicate that the relative freedom from the development of oxidized flavors in the station Jersey herd cannot be attributed to the feeding of mangels.

THE EFFECT OF FEEDING DRIED BEET PULP

The experimental procedure used in the preceding experiments with mangels was followed for the eight Holstein cows of the herd. Quart samples of milk were taken from each cow and processed and handled the same as in the preceding experiment and scored for flavor. A set of samples was taken on November 15 and 17 before any dried beet pulp was fed. Dried beet pulp was fed at the rate of three pounds per cow per feed three times a day, two to two and a half hours before milking to cows number 31 to 34, inclusive, commencing November 20 and was continued throughout the test. Samples were taken November 29 and December 1 while dried beet pulp was being fed.

The average scores of the first two sets of samples are given in Table 4. The milk from the individual cows was uniform in flavor with the exception of the milk from cow number 29 which was more susceptible to the development of oxidized flavor and the milk of cow number 27 which carried a little off-flavor.

As can be seen from Table 5 oxidized flavors developed a little more frequently in the milk from both groups than they did in the earlier tests. However, the flavor of the milk from the four Holsteins receiving dried beet pulp was in no way different from the flavor of the milk from the four not receiving beet pulp.

CONCLUSIONS

From the results of these tests it is concluded that the feeding of mangels or beet pulp in no way prevented or increased the susceptibility of milk to the development of oxidized flavor.

MODIFICATION OF THE BLOOM GELOMETER FOR USE IN THE DETERMINATION OF THE CURD TENSION OF MILK

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After a few simple changes have been made, the Bloom gelometer may be used for the estimation of the curd tension of milk, particularly of those milks which yield a very soft curd on coagulation. The gelometer so modified not only reduces the possibility of a personal factor, but also enables the experimenter to conduct within a relatively short time a large number of tests using the same knife.

DESCRIPTION OF APPARATUS

The Bloom gelometer for the determination of jelly strength was adopted by the Edible Gelatin Manufacturers' Research Society of America in 1923. A detailed description and diagrams of the apparatus were published early in 1924 (4).

A photograph of the modified apparatus together with two of the regular test bottles (4), scales, and the waxed paper cup, used as a receiver for the lead shot, is shown in Figure 1. The receiver weighed only 1.2 grams, since

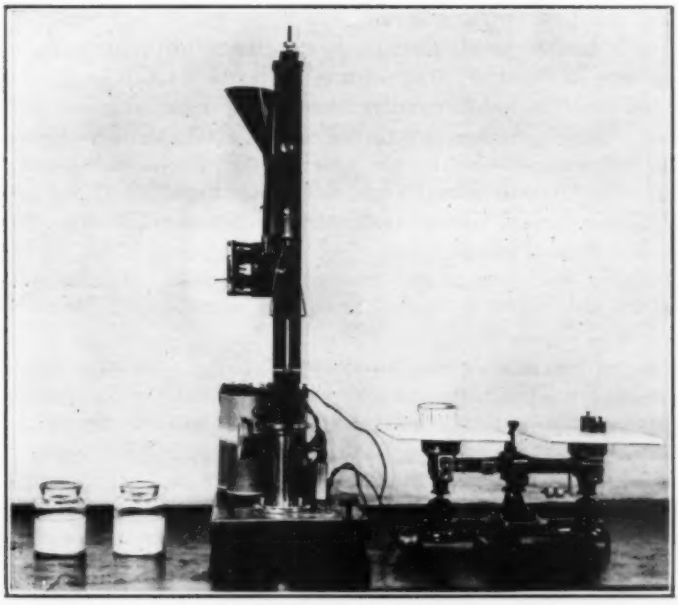

FIG. 1. Modified Bloom Gelometer.

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the weight of a heavy cup might in itself exceed that required to drive the knife through the curd. The rates at which the lead shot were delivered into the receiver at different positions of the dog are recorded in Table 1. The plunger was replaced by a star-shaped knife of the same dimensions as that used with the Hill apparatus (2), so that comparable results might be obtained. The center core of the knife was ground to a needle point extending 10 mm. below the cutting edge. The curd knife, 38 mm. in diameter, was screwed into position with the center needle point and the cutting edges facing downwards. The platform of the gelometer was put on a swivel base so that the test bottle could be placed in position without danger of disturbing the curd. The 4 mm. depression was increased to 17 mm.

EXPERIMENTAL PROCEDURE

The milk was coagulated by the method of Hill (2). Before measuring the curd tension, the disk of the gelometer was adjusted so that it just touched the upper contact point. The test bottle containing the milk curd was lifted carefully from the water bath and placed on a towel to remove surface moisture. The platform of the gelometer was swung out toward the experimenter and the test bottle was brought into position so that the curd knife was suspended within the bottle but did not touch the curd. The platform was then swung back and was slowly raised until the test bottle rested on it and the needle point of the curd knife just touched the surface in the center of the curd. The point of contact was taken as that point at which the tip of the needle point just met the tip reflected in the surface of the curd. The current was now turned on and, after ascertaining once more that the disk was just touching the upper contact, the paper cup was placed on the pan and lead shot was allowed to flow into the cup until the tip of the knife had been driven through the curd for a distance of 17 mm., when the flow of shot stopped automatically. The weight in grams of the cup together with the shot was taken as the observed curd tension. To this value was applied a correction of 11.7 grams, the weight required to drive the knife a distance of 17 mm. in the absence of a curd.

In the earliest experiments we attempted to use a procedure similar to that used in the determination of jelly strength. With the disk touching the lower contact, the needle point of the knife was adjusted as described above. The platform was then raised until the disk just touched the upper contact. But with milks of low curd tension, the curd was too soft to support the weight of the knife, so that cutting through the curd had started before the disk reached the upper contact. The curd tension determination was, therefore, begun with the disk at the upper contact.

The possibility existed that the rate of flow of the shot might affect the curd tension reading. A slight difference in the time required to close the spout would introduce a correspondingly greater error with the dog in the

D_3 position than in the D_1 position. Moreover, the rate at which the knife was driven through the curd might have some influence on the reading. The curd tensions of two different milks were determined with the dog in each of the three positions. The reconstituted milk was prepared by dissolving 12.8 grams of powdered whole milk (Borden's "Parlac" Brand) in 100 cc. of water. The raw whole milk was a sample of Guernsey milk. The curd tensions were also measured with the Hill apparatus (2). The results are given in Table 1.

TABLE 1

The effect of the rate of flow of lead shot from the gelometer on the curd tension reading

Position of dog*	Amount of lead shot delivered in 5 secs.	Curd tension							Curd tension
		Bloom gelometer							Hill apparatus
		Experiment number					Average	Maximum deviation	Average
		(1)	(2)	(3)	(4)	(5)			
	grams			*grams*			*grams*	*grams*	*grams*
				Reconstituted Whole Milk					
D_1	57	13	12	15	14	13	13	3	12
D_2	135	15	15	14	14	15	15	1	
D_3	245	12	17	16	15	14	15	5	
				Raw Whole Milk					
D_1	57	43	42	45	43	41	43	4	58
D_2	135	50	55	46	47	.....	50	9	
D_3	245	55	56	58	60	54	57	6	

* The notations used here: D_1, D_2, D_3, are the same as those used in the description of the apparatus (4). D_1 designates the lowest dog, D_2, the dog directly above it, etc.

The rate of flow of shot apparently made little difference under these experimental conditions in the case of milk of very low curd tension. But with milk of higher curd tension there seemed to be a small but consistent increase in the reading as the knife was driven with increasing speed through the curd. The highest value agreed well with that obtained with the Hill apparatus. In our work on curd tension, measurements were made with the dog in the D_3 position.

The results of a series of measurements with the two types of apparatus are recorded in Table 2. The reconstituted whole milk was prepared as described above. The pasteurized whole milk was purchased from a local store. The raw whole milk was obtained from the afternoon milking of Guernsey

cows and was kept at a low temperature until used the following morning. Each value is the average of from two to five concordant results. In nearly every case the agreement between the results obtained by the two methods and recorded in the third and fourth columns of Table 2 is excellent.

TABLE 2

A comparison of the values obtained for the curd tension of milk with the Hill apparatus and with the modified Bloom gelometer using two kinds of coagulant

Date	Type of milk	Coagulant					
		Pepsin-calcium chloride			Pepsin-hydrochloric acid		
		Hill apparatus	Modified Bloom gelometer	Difference	Hill apparatus	Modified Bloom gelometer	Difference
		grams	*grams*	*grams*	*grams*	*grams*	*grams*
11/17/37	Reconstituted whole milk						
	Sample 1	12	10	+ 2	8	9	− 1
	Sample 2	11	15	− 4	10	11	− 1
11/18/37	Pasteurized whole milk	28	31	− 3	26	16	+10
11/19/37	Pasteurized whole milk	33	32	+ 1	28	16	+12
11/21/37	Raw whole milk						
	Cow No. 2	53	54	− 1	55	18	+37
	Cow No. 6	49	46	+ 3	48	20	+28
	Cow No. 8	45	42	+ 3	43	20	+23
	Cow No. 10	45	36	+ 9	42	12	+30
	Cow No. 12	42	42	0	41	16	+25
	Cow No. 16	37	42	− 5	40	14	+26
	Cow No. 20	47	40	+ 7	40	17	+23
11/22/37	Pasteurized whole milk	35	33	+ 2	25	18	+ 7
11/24/37	Pasteurized whole milk	34	36	− 2			
11/26/37	Raw whole milk						
	Cow No. 1	39	42	− 3			
	Cow No. 2	41	44	− 3			
	Cow No. 3	39	39	0			
	Cow No. 4	45	38	+ 7			
	Cow No. 5	67	61	+ 6			
	Cow No. 6	72	55	+17			
	Cow No. 7	50	52	− 2			
12/3/37	Raw whole milk						
	Cow No. 1	15	18	− 3			
	Cow No. 2	40	39	+ 1			
	Cow No. 3	58	48	+10			
	Cow No. 4	60	68	− 8			
	Cow No. 5	45	49	− 4			
	Cow No. 6	71	70	+ 1			
	Cow No. 7	65	50	+15			
	Average difference			+ 2			+18

COMPARISON OF PEPSIN-HYDROCHLORIC ACID SOLUTIONS WITH THE PEPSIN CALCIUM CHLORIDE SOLUTION AS COAGULANTS IN THE DETERMINATION OF THE CURD TENSION OF MILK

Miller (3) recommended the use of a solution of 0.45 per cent pepsin in 0.4 per cent hydrochloric acid for the coagulation of milk in the determination of curd tension. This coagulant resembles the gastric juice more closely than the Hill coagulant.

In the present investigation milk was coagulated with a solution which

FIGURE-2.

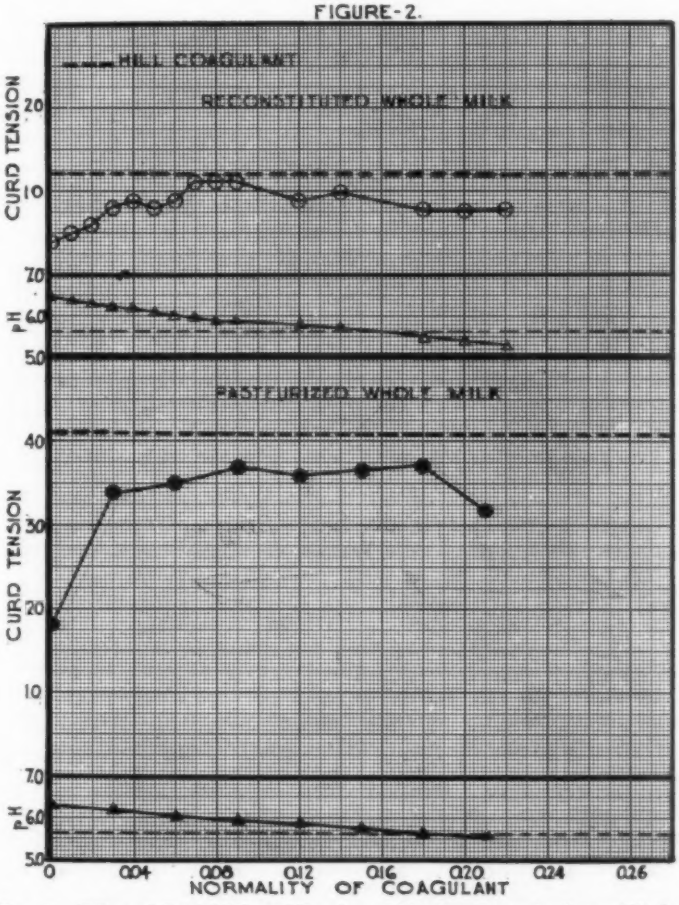

FIG. 2. Effect of the Addition of Pepsin and Varying Concentrations of Hydrochloric Acid on the Curd Tension and the pH of Milk.

contained a constant amount of pepsin and a known but varying amount of hydrochloric acid. The curd hardness was compared with that obtained with the regular Hill coagulant. One cc. of 4.5 per cent pepsin solution and measured quantities of hydrochloric acid of known concentration were pipetted into the test bottles and the volume was diluted with water to 10 cc. During the addition of the 100 cc. of milk the contents of the bottle were given a rotatory motion to ensure complete mixing. After the measurement of curd tension by the Hill apparatus, the curd was thoroughly broken up and the pH of the mixture was estimated with the glass electrode.

The curves of the curd tension and of the pH of two different milks for varying concentrations of hydrochloric acid are given in Figure 2. The addition of pepsin alone to the milk produced a very soft curd. When hydrochloric acid was also used together with the pepsin, the curd tension increased quite rapidly at first and then remained nearly constant over a fairly wide range of pH. The curd tensions of the reconstituted and of the pasteurized milks, which were observed after the addition of the pepsin hydrochloric acid coagulants, were, in general, lower than those obtained with the Hill coagulant. The pH of the milk clot obtained after the addition of the Hill coagulant was lower than that found with the pepsin-hydrochloric acid coagulants, until the concentration of the hydrochloric acid was greater than 0.16 N in the case of the reconstituted milk and about 0.18 N for the pasteurized milks. This agreed well with the observation of Burgwald and Armstrong (1) that the Hill coagulant gave mixtures which were more acid than those formed on the addition of pepsin-hydrochloric acid coagulants, except when the concentration of hydrochloric acid was greater than N/6.67.

The hardness of the curd obtained with the pepsin-calcium chloride coagulant was compared with that produced with a solution containing the same concentration of pepsin in 0.09 N hydrochloric acid. The results are given in the sixth and seventh columns of Table 2. The acid-pepsin coagulant produced a casein curd with a marked tendency toward retraction. With the Hill apparatus the knife tended to lift part of the curd rather than to cut cleanly through it. Greater difficulty was encountered in attempting to measure the hardness of this type of curd with the Bloom gelometer. As the knife passed downward the curd tended to recede, so that for a part of the distance the knife passed through whey rather than curd. As a result the values obtained were much too low. If this type of a curd were to be studied with the Bloom gelometer, it would be necessary to set the knife on the curd itself rather than on the surface of the mixture.

Miller (3) reported that the pepsin-hydrochloric acid coagulant which he used produced a softer curd with boiled milk and a harder curd with raw milk than did the Hill coagulant. However, in our experiments, when the curd tensions of raw milks were measured with the Hill apparatus, the

pepsin-hydrochloric acid coagulant did not give uniformly higher readings than the Hill coagulant.

We wish to thank Dr. Thomas B. Downey for suggesting the use of the gelometer for the estimation of curd tension and for much valuable advice. We are also indebted to Mr. Norman Armstrong for technical assistance.

SUMMARY

The Bloom gelometer has been adapted for use in the estimation of the curd tension of milk. It is especially useful in the measurement of very low curd tensions.

When the regular pepsin-calcium chloride coagulant of Hill is used, the modified gelometer gives results which agree well with those of the Hill apparatus.

If a coagulant consisting of 0.45 per cent of pepsin in 0.09 N hydrochloric acid is used, however, the gelometer under the experimental conditions outlined in this paper gives erroneous results, due, in part at least, to greater retraction of the curd produced by this coagulant.

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SOFT CURD MILK PRODUCED WITH PANCREATIC CONCENTRATE

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The value of soft curd milk in human nutrition has been discussed at length and there have been many and varied attempts and systems of softening the curd of cow's milk. It is generally known that a larger proportion of cow's milk is digested in the stomach of calves in comparison to the amount of human milk digested in the stomach of infants, and it is believed that in order to keep this phenomenon constant these milks are so constituted that this will be possible. The softening of the curd of cow's milk for human consumption is an attempt to make it more nearly like that of human milk with respect to its digestion in infants and adults.

Scales (1) and Carpenter (16) describe three kinds of casein normally present in milk and offer the theory that the change in ratio of these three types of casein may have something to do with the hardness or softness of milk curds. That the digestion of milk is facilitated with the use of soft curd milk is demonstrated by Doan and Welch (2, 3), Espe and Dye (4), and Hill (5). Hill has described the following groups of infants that benefit from the use of soft curd milk: Newly born bottle fed babies, persistent vomiters of whey and leathery curds, colicky babies, the ne'er-do-well group that does not benefit from boiled or sweetened milk, and celiacs and chronic indigestion group and the group that suffers from infantile eczema. Espe and Dye studied the character and activity of the curd in Pavloff pouches of dogs and were able to show that soft curd milk digested more readily than hard curd milk and that the casein content was the factor which affected the nature of the curd more than any of the other milk constituents. Other investigators who discovered that the percentage of casein was very closely related to the type of curd formed in the digestive processes were Scales (1) and The Council on Foods (6), and these investigators generally agree that natural soft curd milk is likewise a low casein milk.

Berry (7) proved that boiling actually lowered the curd tension of milk and also discovered that colostrum was a very hard curd milk. He found that viscolization pressures of 3000 to 5000 pounds were necessary to render hard curd milk soft and this evidence was supported by Theophilus, Hansen and Spencer (8) who decreased the curd tension of milk by homogenization in a single and two-stage homogenizer. Otting and Quilligan (9) described a method of softening the curd by using zeolite sand, during which part of the calcium is removed from the milk thus inhibiting the activity of coagulating enzymes. The Council on Foods (6) also brings out that milk curd may

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be softened by dilution with water, the addition of acids and alkalies and the addition of various cereal extracts.

Lahrmann (10) in making a substitute for mother's milk used digestive ferments. He very nearly completely digested the casein and albumen and added water, sugar, cream, potassium carbonate and phosphoric acid. Von Dungern (11) produced a soft curd milk by coagulating the milk with rennet and then dispersed the curd particles by a mechanical means. Backaus (12) showed that by the addition of alkalies and trypsin to milk in combination with cream and lactose he was able to produce a soft curd milk. Thew (13) produced a soft curd milk by peptonization accompanied by the addition of sodium bicarbonate and condensation. Turney (14) also produced a soft curd milk by the use of a curdling enzyme and concentration of the milk by draining off the whey.

PLAN OF EXPERIMENT

The processes for producing soft curd milk described above have certain disadvantages in that they require special equipment and are in general lengthy procedures. The severe enzyme treatment as recommended by Lahrmann results in the formation of bitter proteoses, peptones and amino acids. Our problem was one of producing a soft curd milk, at a reasonable expense and by a method which would fit into the ordinary dairy plant, without taking away any of the natural nutritive value or minerals of the milk.

The use of digestive enzymes presented itself favorably, in view of the fact that small amounts could be used and there was a possibility that a method could be found for using them in milk plant operations. Preliminary experiments showed that the use of specially prepared (high tryptic, low diastatic) pancreatic enzymes in dilutions of 1–6500 reduced the curd tension considerably but the reaction between the milk protein and the enzyme continued upon the storing of the milk and produced a bitter flavor when the enzyme was added after pasteurization. This brought out the necessity for determining how the enzyme should be inactivated and what the fate of the enzyme plus milk would be under pasteurization conditions.

It was determined that milk could be pasteurized in the presence of pancreatin if the dilution of enzyme with milk was high enough. The inactivation of pancreatin was determined by making dilutions of enzyme with milk and heating the milk to temperatures varying from 62.5 to 73.0° C. The curd softening process was studied from the standpoint of the dilution factor, temperature and time of incubation, type of enzyme used, the storage properties of the softened milk, the effect on the calcium-phosphorus, calcium-magnesium, calcium-protein ratios, formol titration, dialyzability of inorganic material, and the in vivo reaction of the softened milk in calves' stomachs.

METHOD

The method which was used in the curd softening process was devised so that it could be applied with either the flash or the holder method of pasteurization. A typical batch would be prepared by weighing out enough of the pancreatic concentrate to make a dilution of 1–10,000 when incorporated into the milk. This finely ground powder is diluted with enough water so that there are no large particles remaining on the surface and this in turn is poured into the milk to be softened at a temperature of 43° C. The milk is then allowed to incubate for 15 minutes, which is followed by the regular pasteurization process. The temperature used in the flash method was 73.0° C. and the temperature used in the holder was 62.5° C.

The curd tension of the milk was measured by the method of Hill (15) and although this method was found to have various sources of error, each curd tension determination consisted of running four samples and by taking the average of these consistently accurate results were obtained. The data compiled in Table 2 were founded upon the methods recommended by the Association of Official Agricultural Chemists, with the exception of the Walker method which was used in the formol titration.

RESULTS

The results of the inactivation experiments showed that if the milk plus enzyme were held at a temperature of 73° C. for 15 seconds 90 to 95 per cent of the enzyme was inactivated, and if the milk plus enzyme were held at 62.5° C. for ½ hour similar results were obtained. The pasteurization of milk in the presence of pancreatin was possible with dilutions as low as 1–5000.

The data incorporated in Table 1 show that milk curd tension could be lowered consistently to an average of 21.7 grams of tension when the flash and holder method of pasteurization are considered jointly. The decrease in curd tension was accomplished in a variety of ways and with respect to the dilutions used favorable results were obtained with values ranging from 1–6500 to 1–50,000. When the holder method of pasteurization is employed a higher dilution of enzyme can be used. The flash type of pasteurization was used best in conjunction with a 15-minute incubation period at a temperature of 42° C. The time and temperature of incubation required for pasteurization by the holder method was found to have little significance on the amount of reduction in curd tension and this was varied from 24 hours at 7° C. to 15 minutes at 43° C. The average curd tension of the raw milk was approximately 50 grams. Upon storing milk softened by this method it was found that the curd tension decreased an average of 5.6 grams in 72 hours.

The curd tension of milk could be reduced only 5 or 6 grams by the use of diastase when the dilution of diastase was 1–250 and 1–500. These ex-

TABLE 1

Data of soft curd milk produced with variations in dilutions, incubation, and pasteurization

No. of samples	Enzyme used	Dilution in milk	Temp. of incubation °C.	Time of incubation	Temp. of inactivation °C.	Average tension of control	Average tension of softened milk	Average tension after 72 hours
24	Pancreatin	1–6500	36°	15 min.	74° Flash	60 grams	25 grams	23 grams
15	"	1–6500	31°	15 "	76° Flash	50 "	22 "	
15	"	1–10,000	43°	15 "	76° 6 min.	55 "	22 "	
20	"	1–10,000	42°	25 "	76° Flash	50 "	28 "	15 "
15	"	1–20,000	42°	30 "	73° Flash	40 "	28 "	18 "
15	"	1–30,000	42°	30 "	73° Flash	36 "	22 "	26 "
30	"	1–20,000	7°	24 hrs.	62.5° 30 min.	40 "	26 "	20 "
30	"	1–30,000	7°	24 "	62.5° 30 min.	42 "	28 "	24 "
20	"	1–20,000	none	none	62.5° 30 min.	52 "	30 "	21 "
25	"	1–20,000	15°	6 hrs.	62.5° 30 min.	40 "	30 "	24 "
16	"	1–15,000	none	none	62.5° 30 min.	38 "	26 "	24 "
28	"	1–40,000	7°	24 hrs.	62.5° 30 min.	42 "	30 "	22 "
28	"	1–50,000	7°	24 "	62.5° 30 min.	46 "	30 "	24 "

periments on diastase were carried out with the use of a 15-minute incubation period and the flash method of pasteurization.

Other enzymes of vegetable and animal origin such as rennin, pepsin, papain, etc., were used with the same methods of pasteurization and results which were comparable but usually inferior were obtained.

The supernatant fluid obtained from mixing powdered pancreatic concentrate and water was found to have a slight curd softening effect but the decrease in curd tension was far less than that obtained when the dried particles of the pancreatic concentrate were included in the softening material. By allowing the powdered concentrate to soak in water for from one to two hours there was a slight increase in the amount of softening activity but this was found to be of only minor significance.

This method of producing soft curd milk has been tried out on a commercial scale using 100 gallon batches in a large dairy plant and has been found to be practical in all respects. The fat and acidity contents of the milks used were $3.6\% \pm 0.3\%$ and $0.16\% \pm 0.02\%$, respectively. Ammonium carbonate was dissolved in the water into which the pancreatin was mixed and it was found to have an inhibitory action on the curd softening enzymes. No other alkaline materials were used in an attempt to adjust the pH of the water.

Sixteen calves were used in a feeding test, each calf receiving four 1 gallon lots of milk during four feedings. The milk used in the third feeding was colored red and the milk used in the fourth feeding was colored blue so that when the calves were killed seven hours after the last feeding the amount of curd which remained in the stomach from each feeding could be determined. The calves which were fed normal pasteurized milk retained 23 per cent more curd in their stomachs than did the calves which were fed soft curd milk and the tension of the normal curds, as measured by the Hill Curdometer, was twice as great as the enzyme curds. Calves were used in this test because they were thought to be a natural testing medium for cow's milk, and because of the possibility of examining the stomach contents after slaughter.

The data incorporated in Table 2 show the relative value of P_2O_5, protein, calcium, magnesium and formol titration of the control milk in comparison with the enzymatically softened milk and zeolite softened milk. These milks were also dialyzed and the dialysate analyzed for P_2O_5, calcium and magnesium. The whey from the normal and enzyme treated samples was also analyzed for P_2O_5, calcium and magnesium. The P_2O_5 analyses show only small variations. This was found to be true also with the analysis for protein and the formol titration. The results of the magnesium determinations show that probably the only significant feature demonstrated is that the zeolite dialysate samples contained no magnesium. There was such a slight variation in the calcium values of the wheys and dialysates that here

TABLE 2

*Calcium, magnesium, phosphorus, protein and formol titration values of normal, enzyme treated, and zeolite treated milk**

Sample	P_2O_5 %	Ca %	Mg %	Protein (Kjeldahl N × 6.25) %	Formol titration (Walker) (10 cc. sample) cc. N/10 NaOH
Normal Control Milk A	.126	.190	.009	3.13	1.1
Normal Control Milk B	.167	.188	.008	3.07	1.2
Enzyme Softened Milk A	.150	.194	.007	3.02	1.3
Enzyme Softened Milk B	.149	.196	.007	3.00	1.5
Zeolite Milk I	.131	.157	.009	2.97	1.2
Zeolite Milk II	.133	.159	.009	2.72	1.3
Dialysate of Control Milk A	.036	.015	.006		
Dialysate of Control Milk B	.032	.021	.005		
Dialysate of Enzyme Softened Milk A	.038	.026	.004		
Dialysate of Enzyme Softened Milk B	.031	.023	.003		
Dialysate of Zeolite Softened Milk I	.033	.032	.000		
Dialysate of Zeolite Softened Milk II	.038	.043	.000		
Whey of Control Milk A (rennet coagulation)	.088	.070	.007		
Whey of Control Milk B (rennet coagulation)	.087	.065	.005		
Whey of Enzyme Softened Milk A	.089	.075	.009		
Whey of Enzyme Softened Milk B	.090	0.85	.005		

* All samples in duplicate. All "A" and "B" samples represent split batches of the same milk.

too no outstanding significance is demonstrated. The calcium values of the zeolite softened milk samples were found to contain 18 per cent less calcium than the control samples and the enzyme treated samples.

Clinical investigations on a milk prepared by this method are being carried out.

CONCLUSIONS

The curd tension of ordinary cow's milk may be softened by pancreatic concentrate to within a range of 20 to 30 grams and the curd tension of samples of milk which have been softened enzymatically decrease in their curd tension value upon storage.

Either the flash or holder type of pasteurization may be employed in the method described above.

Calves fed milk which has been softened with pancreatic concentrate retain the curd in their stomachs for a shorter period of time and the curd formed therein is much softer than the curd formed when normal pasteurized milk is fed.

The P_2O_5, calcium, magnesium, protein and formol titration values are changed so slightly from that of pasteurized milk that they are of little significance in studying the nature of enzymatically softened milk.

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THE CHEMICAL COMPOSITION AND PROPERTIES OF NORMAL AND RANCID JERSEY MILK

III. TITRATABLE ACIDITY, HYDROGEN-ION CONCENTRATION AND LIPASE CONTENT

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A study has been made of the chemical composition of the milk of a Jersey herd in relation to the off-flavor, rancidity. In the course of the study normal values were established for several constituents of milk, both for individuals and for the herd throughout the entire lactation period. The composition of rancid milk has been compared with that of normal milk of the same individual and of the herd. The chloride, lactose, fat, total solids and protein content of the milk has been reported in previous papers (3) (4). The present paper discusses the titratable acidity, hydrogen-ion concentration and lipase content of normal and rancid milk.

Published data indicate a wide range in the titratable acidity and pH of normal milk. This variation is attributed to such factors as breed, environment, period of lactation, and individuality. In the present study the number of variable factors was reduced by employing a single breed of cows, by maintaining them under the same environmental conditions and by comparing the composition of rancid milk with that of normal milk produced in the same period of lactation.

EXPERIMENTAL

Management of animals and methods of obtaining milk samples have been described in a previous paper (3). Over a period of eight months, determinations were made of the titratable acidity and pH values of samples taken weekly from each of 18 members of the Jersey herd. Lipase determinations were made over a period of 20 months.

Titratable acidity was determined by titrating 10 cc. of the fresh sample with 0.1 N NaOH, using phenolphthalein as an indicator. The degree of acidity of the sample is expressed as the percentage of lactic acid. pH was determined by use of the quinhydrone electrode. Methods used in estimation of the lipolytic activity of milk are discussed later.

PRESENTATION OF DATA

Titratable Acidity and Hydrogen-Ion Concentration of Normal Jersey Milk

The mean titratable acidity and pH values of all milk samples obtained from each of 12 animals are shown in Table 1. The mean acidities ranged

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from 0.158 per cent to 0.112 per cent; individual samples showed minimal and maximal values of 0.122 per cent and 0.410 per cent, respectively. The mean pH values ranged from 6.60 to 6.43, with minimal and maximal values of 6.56 and 6.10 for individual samples.

Monthly changes in the mean titratable acidity and pH values of the normal milk of the herd are shown in graphs 1 and 2, Figure 1. The general

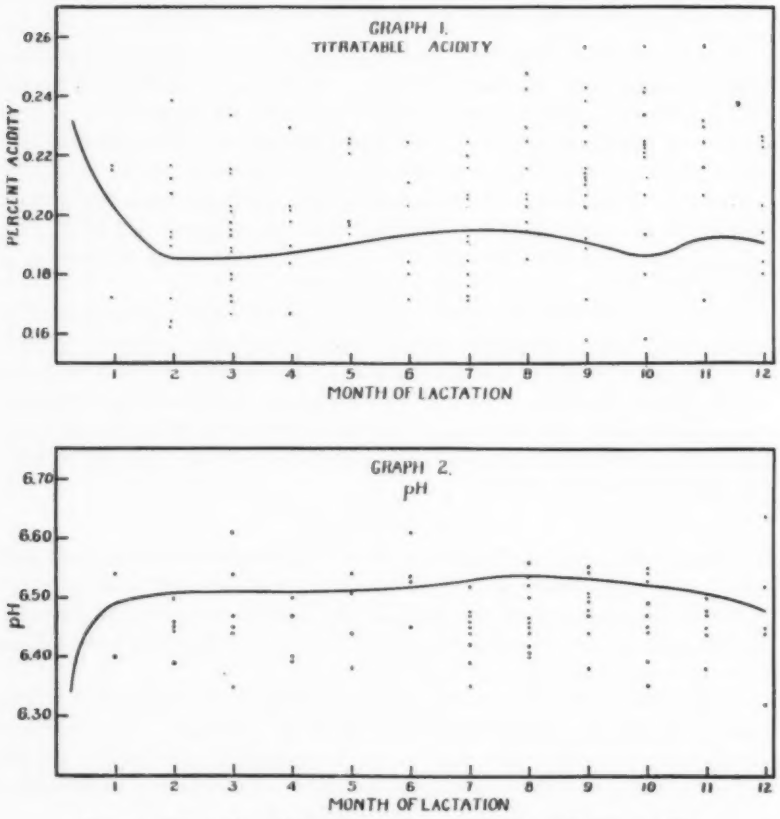

FIG. 1. Titratable acidity and pH of normal and rancid Jersey milk.

Graph 1

—— Mean titratable acidity of all normal samples for each month of the lactation period.
● Titratable acidity of rancid samples.

Graph 2

—— Mean pH of all normal samples for each month of the lactation period.
● pH of rancid samples.

trends of titratable acidity and hydrogen-ion concentration throughout lactation were similar, both showing a rapid decrease at the beginning of lactation, with little change thereafter. During the first six weeks the mean titratable acidity dropped from 0.234 per cent to 0.185 per cent, and the pH increased from 6.34 to 6.53, after which they remained nearly constant until the end of lactation.

The above data are summarized in Table 2, which shows the mean titratable acidity, hydrogen-ion concentration and pH of all normal samples for each of twelve four-week periods.The coefficients of variation indicate a marked variation in milk from different animals in the same period of lactation. The mean titratable acidity of 357 normal samples was 0.189 ± 0.004 per cent with a standard deviation of ± 0.027, and a coefficient of variation of 20.6; the mean hydrogen-ion concentration was $3.05 \pm 0.04 \times 10^{-7}$ (pH 6.52) with a standard deviation of ± 0.63 and a coefficient of variation of 14.2.

Caulfield and Riddell (1) have reported values for the titratable acidity of the milk of six Jersey cows as determined by samples collected at monthly intervals throughout a complete lactation. Their values are in close agreement with those reported here. They found an average titratable acidity of 0.179 per cent, individual variation in average acidities of from 0.098 per cent to 0.295 per cent, and a range of from 0.194 per cent in the first month to 0.145 per cent in the tenth month of lactation. They also observed a marked drop in titratable acidity between the first and second months of lactation, after which the acidity remained nearly constant through the seventh month. During the last month of lactation they found a marked decline in the average acidity of milk. Such a decline was not observed in the present study; instead, a slight increase in titratable acidity manifested itself in the milk of animals whose lactation period was unduly prolonged.

The fairly wide deviation in the pH of milk is well recognized. Sommer (5) in a recent study observed a range of from 6.77 to 6.22 in the pH values of 386 milk samples obtained from 43 cows (breed not specified). 140 of the samples had pH values of from 6.36 to 6.45 and 144 from 6.46 to 6.55. These values are in agreement with those observed in the present study.

Titratable Acidity and Hydrogen-Ion Concentration of Rancid Jersey Milk

As may be seen in Table 1, five animals produced no rancid milk during the period in which titratable acidity and pH were determined. The mean titratable acidity of their milk varied from 0.158 per cent to 0.203 per cent, with an average value of 0.185 per cent; the pH values ranged from 6.60 to 6.47, with an average of 6.54. Seven animals produced rancid milk comprising 19 to 100 per cent of all samples obtained from them. The mean titratable acidity of their milk ranged from 0.162 per cent to 0.212 per cent and averaged 0.194 per cent; the pH values varied from 6.53 to 6.43, with

an average of 6.48. In considering the milk of the individual animals, only in the case of cows 6 and 11 was the average titratable acidity of rancid samples appreciably higher than that of the normal milk. The pH of the rancid milk of animals 6, 9 and 10 was appreciably lower than that of their normal milk.

To facilitate comparison with normal values for the period of lactation in which they occurred, the titratable acidity and pH values of rancid samples are indicated as separate points on the graphs in Figure 1. From the graphs it is evident that rancid samples tended to have higher titratable acidities and lower pH values than did normal milk of the same period of lactation.

In Table 3 rancid samples are grouped according to their degree of rancidity. Samples described as "very slight" or "doubtfully rancid" had practically the same mean titratable acidity and hydrogen-ion concentration as did normal milk. Those criticized as "very rancid," "rancid," and "slightly rancid" had a mean acidity of 0.212 per cent as compared to 0.189 per cent, the mean acidity of all normal samples; their mean pH was 6.41 as compared with 6.51, the mean value for normal milk. Both the mean titratable acidity and the hydrogen-ion concentration of all rancid samples were significantly higher than the mean values of all normal samples.

METHODS OF ANALYSIS

Hileman and Courtney (2) have recently reviewed previous work on the lipase content of milk. In the present study an attempt has been made to secure a method capable of detecting the presence of small amounts of lipase and to provide a standard with which the lipolytic activity of milk might be compared.

Two methods of estimation were employed in the study of the lipase content of milk. The first was an adaptation of the general procedure of Willstätter, Waldschmidt-Leitz and Memmen (6). The lipolytic activity of a known volume of milk was estimated by determining the amounts of free fatty acid liberated by the hydrolysis of olive oil in the presence of an accelerator. Determinations were made in the following manner. 2.5 gms. of olive oil, 2 ml. of an ammonium chloride-ammonium hydroxide buffer, pH 8.9, 1 ml. of a one per cent calcium chloride solution and 10 ml. of milk were shaken vigorously for 3 minutes, and then placed in a water bath at 30° C. At the end of the incubation period the contents of the flask were washed into a 250 ml. Erlenmeyer flask with 112 ml. of 95 per cent alcohol. Twenty ml. of ether and 1 ml. of a saturated solution of potassium oxalate were added and the fatty acids titrated with N/10 alcoholic NaOH, thymolphthalein being used as an indicator. The amount of acid produced was determined by subtracting the initial acidity of the reaction mixture, determined in controls, from the acidity of the reaction mixture following incubation.

To ascertain the minimum amount of lipase detectable by this method,

determinations were made of the degree of hydrolysis produced by decreasing amounts of a commercial preparation of lipase during a one hour's incubation period. In the first series of experiments an aqueous medium was employed. The addition of 240, 160, 100, 80, 60 and 40 mgs. of the lipase preparation produced amounts of acid equivalent to 18.0, 15.0, 10.0, 8.0, 6.5 and 4 ml. of 0.1 N NaOh, respectively; the lipolytic activity of amounts less than 20 mgs. is shown by curve 1, Figure 2.

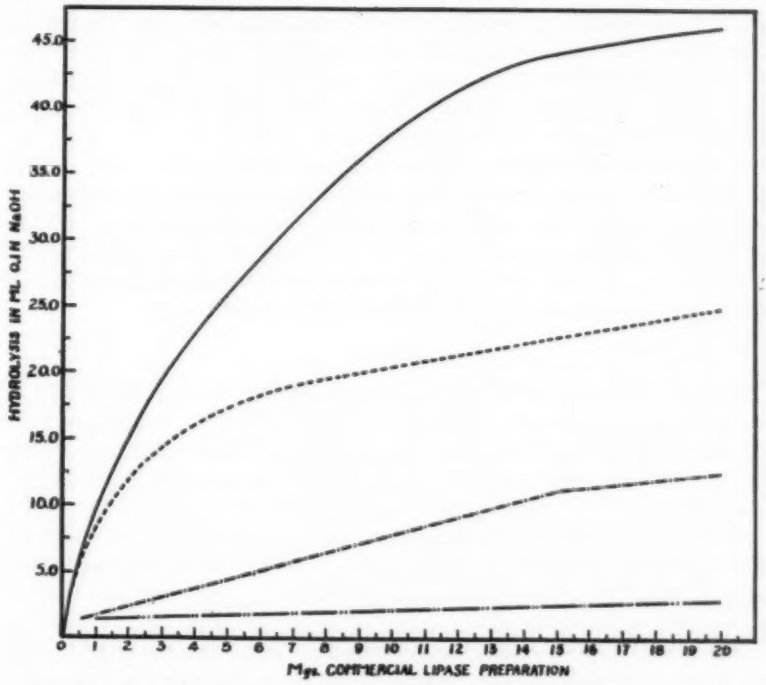

FIG. 2. Hydrolysis of olive oil and tributyrin by commercial lipase preparation in media of milk and water.

·——· Aqueous medium, olive oil substrate, 1 hour incubation.
·—·—· Milk medium, olive oil substrate, 1 hour incubation.
– – – – Milk medium, olive oil substrate, 24 hours incubation.
——— Milk medium, tributyrin substrate, 24 hours incubation.

In the second series of experiments, inactivated milk served as the medium. Inactivation was effected by heating the milk for one hour at 70° C. Curve 2, Figure 2, shows the degree of hydrolysis produced in one hour by amounts of lipase preparation ranging from 20 mgs. to 0.6 mgs.

The emulsion of milk and olive oil proved to be more favorable to the action of lipase than the emulsion of water and olive oil. By increasing the

period of incubation from one to 24 hours, it was possible, using milk as a dilutant, to detect the presence of 0.06 mg. lipase preparation, as may be seen from curve 3, Figure 2. A 24-hour incubation period was therefore adopted.

In a second method later employed for the estimation of lipase, two 50 ml. aliquots of each milk sample were used, one aliquot serving as a control. The control was heated for one hour at 70° C. to inactivate the lipase present. To each of the aliquots were added 50 ml. water, 0.5 ml. tributyrin and 4 drops of formaldehyde. The two reaction mixtures were then shaken for three minutes and incubated for 24 hours at 30° C., after which they were diluted and titrated as described above. The difference between the titration values of the raw and inactivated samples after incubation was taken as a measure of the degree of hydrolysis produced.

To determine the smallest amount of lipase which could be detected by this method, known amounts of lipase preparation were added to previously inactivated milk. Lipase added to the controls was also inactivated. Results obtained by this method are given in Curve 4, Figure 2. This method proved as sensitive as the first method for amounts of the lipase preparation less than 1 mg. and more sensitive for amounts greater than 1 mg.

RESULTS

The two methods described above were used in estimating the lipase content of 490 samples of milk, 121 of which were rancid. The results are given in Table 4. In this table samples are grouped according to flavor and without regard to the period of lactation, since there appeared to be no correlation between the period of lactation and the amount of lipase present. Table 4 also includes the amount of commercial lipase preparation which under the same conditions produced a corresponding degree of hydrolysis. These amounts were read from curves 3 and 4, Figure 2, when methods 1 and 2 respectively, were employed.

The results indicate that all samples had a definite lipolytic action which was, however, very small when compared with a commercial lipase preparation. As determined by the first method, 10 ml. of normal milk produced the same degree of hydrolysis as did approximately 0.15 mgs. of the lipase preparation; when the second method was employed 50 ml. of normal milk showed a lipolytic activity equivalent to that of 0.4 mg. of lipase preparation.

By both methods, samples described as "very slightly" or "doubtfully" rancid were found to have a lipolytic activity approximately equal to or somewhat less than that of normal milk; "slightly" rancid samples produced a somewhat greater amount of acid than did normal milk, differences in the average titrations being 0.8 mg. and 0.9 mg. for the two methods. Twenty-seven "very rancid" and "rancid" samples analyzed by the first procedure showed about twice as much lipolytic activity as normal milk. One "very

TABLE 1

Titratable acidity and pH values of normal and rancid milk of 12 Jersey cows

Cow number	Milk samples		Mean titratable[1] acidity			Mean pH		
	Normal	Rancid	Normal	Rancid	Total	Normal	Rancid	Total
	number	*number*						
1	20	0	0.176	.....	0.176	6.55	.....	6.55
2	35	0	0.158	.....	0.158	6.60	.....	6.60
3	29	0	0.203	.....	0.203	6.51	.....	6.51
4	18	0	0.189	.....	0.189	6.56	.....	6.56
5	27	0	0.203	.....	0.203	6.47	.....	6.47
6	21	4	0.198	0.275	0.198	6.52	6.33	6.47
7	24	4	0.203	0.203	0.203	6.51	6.47	6.51
8	15	7	0.176	0.185	0.180	6.49	6.53	6.50
9	22	9	0.212	0.216	0.212	6.46	6.35	6.43
10	9	17	0.162	0.167	0.162	6.57	6.46	6.53
11	14	18	0.194	0.225	0.212	6.47	6.47	6.47
12	0	30	.....	0.203	0.203	.....	6.48	6.48

[1] Expressed as percent lactic acid.

TABLE 2

Titratable acidity and hydrogen-ion concentration of normal[1] Jersey milk in relation to the period of lactation

Lactation periods	Milk samples	Titratable acidity[2]			Hydrogen-ion concentration ($\times 10^{-7}$)			pH[6]
		Mean	S. D.[3]	C. V.[4]	Mean	S. D.[3]	C. V.[4]	
weeks	*number*			*percent*			*percent*	
1–4	40	0.203 ± 0.005[5]	0.035	17.3	3.68 ± 0.13[5]	0.84	22.8	6.43
5–8	48	0.185 ± 0.004	0.024	12.9	3.05 ± 0.08	0.54	17.6	6.51
9–12	27	0.185 ± 0.005	0.022	11.7	3.07 ± 0.07	0.49	16.0	6.51
13–16	36	0.194 ± 0.005	0.030	15.0	2.96 ± 0.05	0.35	12.0	6.53
17–20	32	0.185 ± 0.005	0.029	15.7	3.01 ± 0.11	0.74	24.5	6.52
21–24	30	0.194 ± 0.005	0.031	15.6	3.10 ± 0.09	0.57	18.3	6.51
25–28	22	0.194 ± 0.005	0.022	11.1	2.92 ± 0.06	0.42	14.5	6.53
29–32	26	0.194 ± 0.006	0.029	11.1	2.89 ± 0.06	0.41	14.3	6.54
33–36	28	0.185 ± 0.005	0.023	15.0	2.74 ± 0.07	0.45	16.4	6.56
37–40	14	0.180 ± 0.006	0.023	12.7	3.07 ± 0.08	0.55	17.9	6.51
41–44	10	0.194 ± 0.005	0.024	12.8	3.01 ± 0.07	0.45	15.2	6.52
45–48	13	0.185 ± 0.006	0.023	11.7	3.29 ± 0.18	1.19	36.0	6.48

[1] No rancid samples are included.
[2] Expressed as percent lactic acid.
[3] Standard deviation of mean.
[4] Coefficient of variation of mean.
[5] Standard error of mean.
[6] Corresponding to the mean hydrogen-ion concentration for the period.

rancid'' sample analyzed by the second method produced approximately ten times as much acid as did normal milk. This was, however, the only sample to display such a marked degree of activity.

One would conclude, therefore, that all milk contains lipolytically active

TABLE 3

Titratable acidity and hydrogen-ion concentration of normal and rancid Jersey milk

Description of samples	Milk samples	Titratable acidity[1]		Hydrogen-ion concentration ($\times 10^{-7}$)			pH[4]
		Mean	Standard deviation	Milk samples	Mean	Standard deviation	
	number			*number*			
Very rancid, rancid and slightly rancid	84	0.212 ± 0.005[2]	0.035	56	3.89 ± 0.17	1.30	6.41
Very slightly and doubtfully[3] rancid	47	0.194 ± 0.004	0.023	21	3.20 ± 0.09	0.48	6.49
All rancid samples	131	0.203 ± 0.003	0.033	77	3.68 ± 0.09	1.17	6.43
Normal	357	0.189 ± 0.002	0.027	211	3.05 ± 0.04	0.63	6.51

[1] Expressed as percent lactic acid.

[2] Standard error of mean.

[3] Samples criticized as rancid by less than half the judges.

[4] Corresponding to the mean hydrogen-ion concentration.

TABLE 4

The lipolytic activity[1] of normal and rancid Jersey milk

Description of samples	Method 1. (10 ml. milk)			Method 2. (50 ml. milk)		
	Milk samples	Hydrolysis in ml. 0.1 N NaOH	Commercial lipase equivalent[2]	Milk samples	Hydrolysis in ml. 0.1 N NaOH	Commercial lipase equivalent[2]
	number		*mg.*	*number*		*mg.*
Very rancid	1	4.2	0.40	1	43.6	14.6
Rancid	26	2.8	0.25	3	9.7	1.0
Slightly rancid	32	2.3	0.20	25	5.4	0.5
Very slightly rancid	10	1.5	0.15	9	3.9	0.4
Doubtfully[3] rancid	13	0.9	0.10	5	3.8	0.4
Normal	159	1.5	0.15	210	4.5	0.4

[1] Expressed in terms of ml. 0.1 N NaOH required to neutralize the free fatty acids produced during a 24-hour incubation period.

[2] Amount of commercial lipase preparation producing a degree of hydrolysis equal to that produced by milk samples under the same conditions.

[3] Samples criticized as rancid by less than half the judges.

substance and that milk which has a definitely rancid flavor is somewhat more active lipolytically than is normal milk.

SUMMARY AND CONCLUSIONS

Data have been presented showing the titratable acidity, hydrogen-ion concentration and lipase content of the milk of animals of a Jersey herd, all of which received the same ration and were subject to the same environmental conditions. The amounts found in milk criticized as rancid have been compared with those present in normal milk of the same period of lactation.

Rancid milk was found usually to have a higher titratable acidity and hydrogen-ion concentration than normal milk of the same period of lactation. The mean titratable acidity and hydrogen-ion concentration of all rancid samples were significantly higher than the mean values for all normal samples.

All milk was found to contain a small amount of lipolytically active substance. Definitely rancid milk was somewhat more active lipolytically than was normal milk.

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A COMPARATIVE STUDY OF METHODS OF DETERMINING THE MOISTURE CONTENT OF CHEDDAR CHEESE

II. THE STEAM OVEN METHOD AT HIGH PRESSURE AND THE OLIVE OIL METHODS*

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INTRODUCTION

In a previous study (1) a discussion of the use of an open flame-olive oil method for the determination of moisture in Cheddar cheese was given and its accuracy compared with the Mojonnier method, slightly modified. In addition, a modification of the oil method was suggested in which a small amount of sodium chloride was added to the oil. The addition of the salt prevented the cheese from lumping and sticking during heating, and also prevented the cheese from spattering. The olive oil method gave results approximately 0.3 per cent above those secured by the Mojonnier method, whereas the oil method, modified by the addition of salt, gave results averaging approximately 0.55 per cent higher than the results by the Mojonnier procedure.

The steam pressure oven method for moisture determination was originally developed for butter (2), but has since been more widely adopted for cheese analysis. Sammis (3) (4) gives in detail the steps involved in the moisture determination of cheese by this method, and points out that a 10 gram sample is dried satisfactorily in at least 4 to 5 hours and a 5 gram sample in about one-half of this time when the steam pressure ranged from 50 to 60 pounds. According to this author, excessive heating periods in the steam pressure oven had little effect on new cheese, but old cured cheese continued to lose weight at a noticeable rate. Van Slyke and Price (5) point out that disastrous effects on the samples may occur if the oven is connected directly with a boiler carrying a high steam pressure without having an intervening valve. Sammis (3) in discussing the steam pressure oven method, states that "duplicates commonly agree with each other within 0.5 per cent."

In connection with the study of the olive oil method previously reported (1), samples of Cheddar cheese which were analyzed for moisture by the oil procedure were also dried to constant weight by the use of a steam pressure oven. It was thought desirable to tabulate the data collected and to compare the results obtained by these methods.

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EXPERIMENTAL

The cheese was prepared and kept for analysis according to directions previously outlined (1). The moisture analysis by use of the steam pressure oven was made on a 2–3 gram sample which was weighed in an aluminum dish approximately 55 mm. in diameter, 22 mm. in height, and provided with a slip-in inverted cover fitting tightly on the inside. The drying was carried out in a Farrington steam pressure oven connected directly to the main line which carried a steam pressure of approximately 85 pounds. Two and one-half hours were usually required to bring the samples to constant weight.

Analysis of the cheese by the olive oil method was carried out (a) by use of a 5 gram sample of finely chopped cheese in 20 cc. of olive oil; and (b) by use of 5 grams of cheese in 20 cc. of olive oil which contained approximately 1 gram of sodium chloride. The latter procedure will be referred to as the modified olive oil method. More detailed information concerning the procedure for the oil methods has been given (1).

Differences between Duplicates: Excellent checks between duplicate determinations were obtained by the steam oven method. The average difference between duplicates of 29 trials by this method was $0.19 \pm .02$ per cent with 18 or approximately 62 per cent of the duplicates varying 0.2 per cent or less. Only one of the 29 trials showed variation between duplicates greater than 0.5 per cent. The variations one might expect by the use of the olive oil method were discussed previously (1) in which it was found that in 28 trials, the average difference between duplicates by the regular oil method was found to be $0.26 \pm .03$ per cent. Sixty-four per cent of these duplicates varied not greater than 0.2 per cent. In the case of the modified olive oil method, 63 per cent of the duplicates gave variations which fell within this range, with the average difference of 20 trials being $0.20 \pm .03$ per cent.

Comparison of Results: The results of the analysis of Cheddar cheese by the steam pressure oven method, by the olive oil method, and by the modified olive oil method are given in Table 1.

The results show the steam pressure oven method, at the high pressure operated, gave results averaging approximately 0.3 per cent above those secured by the regular olive oil method. The average of the individual differences for the 31 trials was somewhat greater, averaging $0.49 \pm .04$ per cent. That the oven method tends to give higher results is shown by the fact that 28 of the 31 trials gave higher values by the steam oven method than by the regular olive oil procedure.

The modified olive oil method gave results which for 17 trials averaged within 0.1 per cent of the steam oven method. The averages of the differences between the results for the individual trials was $0.31 \pm .04$ per cent. Ten of the moisture analyses were lower by the modified oil method, whereas

TABLE 1

The moisture content of Cheddar cheese when determined by the steam oven method at 85 pounds pressure, by the olive oil method, and by the modified olive oil method

Sample	Steam Pressure Oven Method	Olive oil method		Modified oil method	
No.	Moisture	Moisture	Difference from steam oven	Moisture	Difference from steam oven
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*
1	37.75	36.60	− 1.15	.....	.....
2	35.53	34.90	− 0.63	.....	.....
3	34.52	34.31	− 0.21	.....	.....
4	34.69	33.85	− 0.84	.....	.....
5	34.53	34.14	− 0.39	.....	.....
6	34.48	33.75	− 0.73	.....	.....
7	34.85	34.03	− 0.82	.....	.....
8	36.63	35.72	− 0.91	.....	.....
9	30.69	29.99	− 0.70	.....	.....
10	33.92	33.87	− 0.05	.....	.....
11	31.73	30.29	− 1.44	.....	.....
12	32.49	31.73	− 0.76	.....	.....
13	44.62	44.02	− 0.60	.....	.....
14	44.24	44.05	− 0.19	.....	.....
15	44.08	43.50	− 0.58	.....	.....
16	42.82	42.76	− 0.06	.....	.....
17	41.94	41.96	+ 0.02	.....	.....
18	43.81	43.77	− 0.04	.....	.....
19	38.86	38.71	− 0.15	.....	.....
20*	38.34	.....	.....	38.07	− 0.03
21	36.95	36.43	− 0.52	36.76	− 0.19
22	37.02	36.75	− 0.27	37.17	+ 0.15
23	33.78	32.78	− 1.00	33.10	− 0.68
24	33.83	33.54	− 0.29	33.12	− 0.71
25	34.44	34.02	− 0.42	34.48	+ 0.04
26	37.54	37.28	− 0.26	37.45	− 0.09
27*	38.80	.....	.....	38.89	+ 0.09
28	40.86	40.28	− 0.58	40.84	+ 0.04
29*	40.75	.....	.....	40.60	− 0.15
30	35.10	34.76	− 0.34	34.83	− 0.27
31*	34.57	.....	.....	34.85	+ 0.28
32	36.37	36.46	+ 0.09	.....	.....
33	39.52	40.24	+ 0.72	39.89	+ 0.37
34	35.41	35.23	− 0.18	34.84	− 0.57
35*	39.89	.....	.....	39.61	− 0.28
36*	40.95	.....	.....	41.91	+ 0.96
37	39.94	39.75	− 0.19	39.77	− 0.17
Average, 31 trials	37.19 ± .47	36.75 ± .49	0.49 ± .04**	.....	.....
Average, 17 trials	37.51 ± .41	.....	.....	37.42 ± .45	0.31 ± .04**

* Samples spattered when attempts were made to analyze them by the regular olive oil procedure.

** Signs neglected in computing average.

7 were higher. As was observed in the previous paper, certain samples analyzed by the modified olive oil method could not be analyzed by the regular oil procedure due to spattering during heating which threw a portion of the material from the dish.

SUMMARY

A comparison was made between the steam oven method operating at approximately 85 pounds pressure; the regular olive oil method, in which 5 grams of cheese are placed in olive oil and heated to dryness directly over a small gas flame; and the modified olive oil in which sodium chloride is added to the cheese-olive oil mixture to prevent spattering and sticking of the cheese. The results showed the olive oil method to give values averaging approximately 0.35 per cent lower than the oven method, whereas the salt-olive oil procedure gave results which averaged within 0.1 per cent of those secured by the steam oven.

Either of these oil methods appear to give results which, on the basis of these comparisons with the steam pressure oven method, would be accurate enough for all practical purposes. Further, the oil methods have the distinct advantage of requiring less time than the oven procedure, since the test may be completed within 20 to 30 minutes, especially when the modified procedure is followed.

The modified oil method has greater applicability than the regular olive oil method since (a) it permits more rapid heating and drying due to the fact that the particles of cheese do not lump together, (b) it prevents the cheese from sticking to the bottom of the pan during the heating process, and (c) it has permitted practically all of the Cheddar cheese samples thus far encountered to be analyzed without experiencing the difficulty with spattering which occurs frequently with the regular oil method. However, during some recent trials, two samples of cheese did not lend themselves to moisture determination by the modified oil method because of spattering. Both of these samples were abnormally high in moisture which may account for their behavior.

Although this and the previous paper have dealt with the use of olive oil in the open flame test, other oils with higher volatilization points may be superior to the olive oil for this method of moisture analysis. It was observed in the trials reported that some volatilization of the olive oil did occur during heating, resulting in the formation of a disagreeable odor. This slight volatilization, however, did not appear to have any appreciable influence on the moisture results. Several trials have since been carried out using cottonseed oil, and indications are that this oil may be superior to olive oil for the moisture test. It has a higher volatilization point and is considerably less expensive. Mineral oil is less satisfactory since it requires a relatively low temperature for volatilization.

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THE KEEPING QUALITY OF BUTTERS

I. THE RATES OF DETERIORATION OF BUTTERS MADE FROM CREAMS OF DIFFERENT ACIDITIES AND STORED AT VARIOUS TEMPERATURES*

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The question of the effect of the acidity of cream upon the keeping quality of its butter has been the subject of numerous discussions and investigations. While it is generally agreed that butter made from sweet cream keeps better in storage than butter made from acid cream, there seems to be some differences of opinion on this subject when dealing with a product made from cream known as "low-acid cream." Furthermore, all acid cream butters do not exhibit the same rate of deterioration in storage, but vary in this respect with the degree of acidity of the cream from which they were prepared.

In reports of the Iowa Experiment Station (1) (2) (1890 and 1892) work is reported which showed that butter made from sweet cream kept better than did butter made from sour cream. In each instance raw cream was used. Studies begun in 1905 by the U. S. Department of Agriculture (3) (4) (5) of the influence of the acidity of cream upon the keeping quality of butter, established the fact that butter made from unripened pasteurized sweet cream maintained its fine quality to a high degree during at least 8 months of storage at 0° C.

The deterioration of butters made from creams of different acidities was studied by White, Trimble and Wilson (6), who found that butters made from creams with acidities of from 0.15 per cent to 0.31 per cent kept well in storage at 0° F. for 8 months. After 12 months at 0° F. butters from creams of from 0.15 to 0.25 per cent acidity had deteriorated less than those made from creams of 0.28 to 0.31 per cent acidity, and the latter had deteriorated less than those made from creams of higher acidities. They found also that ripening cream with a lactic culture, even to relatively low acidities improved the score of the fresh butter therefrom, but the improvement was usually lost in storage. The deterioration was judged entirely by loss of score. The results upon the butters from cultured cream confirmed the conclusions of Mortensen (1922) (7) that ripened cream butter received a higher commercial score than did sweet cream butter when fresh but that sweet cream butter kept better in storage. This author states further (1936) (8):

"Research on cream ripening was continued for several years and we recommend at present that the acidity shall not exceed .36 per cent in the

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serum. That represents an acidity in a 30 per cent cream of .70 × .36 or .25 per cent. Whether this figure will be changed somewhat in the future will depend on the research of the next few years; it is my belief that it is about right. By applying the proper system of ripening, this degree of acidity will produce flavor and keeping qualities superior to those of sweet cream butter."

In order to determine accurately the relative keeping quality of butters from creams of different acidities their rates of deterioration over a relatively long storage period must be known. This is especially true of butters made from low acid creams whose rates of deterioration are slow in the initial periods of storage and accelerate with increased time of storage. In the present work, therefore, samples of each butter were, except in a few cases, scored and tested at regular intervals until the respective butter was declared unfit for consumption.

Though "score" has proved invaluable as an index of the quality of butter it is not especially valuable as a test by which chemical changes of butter may be detected or followed with accuracy. It is rather an opinion of quality based upon a number of factors and has no definite meaning except as it relates to edibility of the product. In the present work the "score" has been supplemented with chemical tests which have proved of value in the study of the deterioration of fats and oils. These tests have furnished data of a quantitative nature by means of which the rate of deterioration through oxidation changes have been accurately expressed and the relative value of the score method and other methods determined. The data obtained furnished also some information upon one of the major problems under consideration; namely, a method which would indicate early in the storage period the susceptibility of butterfat to chemical change and could therefore be used to predict keeping quality.

EXPERIMENTAL

Fresh creams of 0.12 to 0.14 per cent acidity were pasteurized and churned to furnish the control samples of butter. Other samples were pasteurized, brought to an acidity of approximately 0.20 per cent, others to acidities of approximately 0.30 and 0.40 per cent, respectively, and then churned. The butters from each churning were divided into five lots and samples of these were stored at each of five different temperatures. The chosen temperatures of storage were 20° C., 10° C., 0° C., – 10° C., and – 17° C., respectively. The samples stored at – 17° C. were tested and scored at 90-day intervals, those at – 10° C. at 80-day intervals, etc., as indicated in Table I.

The tests chosen to indicate the rate of deterioration were score, peroxide value, time of bleaching at 42° C., and two dye reduction tests. The last four of these tests are definitely related to oxidation, a reaction which our studies on fats and oils have led us to believe is directly and indirectly an underlying cause for many of the off flavors of butter. Because of many

TABLE 1

The averages of values obtained in the different tests, upon butters stored for varying periods of time at different temperatures

Average acidity of cream (% lactic)	Number of samples	Days in storage	Peroxide value (M. mols per k)	Bleaching time at 42° C. (Days)	Average score	Days in storage	Peroxide value (M. mols per k)	Bleaching time at 42° C. (Days)	Average score	Days in storage	Peroxide value (M. mols per k)	Bleaching time at 42° C. (Days)	Average score	Days in storage	Peroxide value (M. mols per k)	Bleaching time at 42° C. (Days)	Average score	Days in storage	Peroxide value (M. mols per k)	Bleaching time at 42° C. (Days)	Average score
Storage at 20° C.																					
.13	5	0	0	88	91.7	20	0.18	.....	89.6	52	0.88	66	88.7								
.19	8	0	0	86	91.7	20	.09	.....	89.8	52	.73	71	89.0								
.31	5	0	0	88	92.0	20	.62	.....	89.0	52	1.62	31	88.2								
.41	4	0	0	70	91.25	20	1.25	.....	86.6	52	7.41	13	86.3								
Storage at 10° C.																					
.13	5	0	0	88	91.7	35	0.28	90	91.1	72	0.63	79	90.0	108	1.26	46	89.1				
.19	8	0	0	86	91.7	35	.19	102	89.8	72	.61	71	89.7	108	1.21	51	89.4				
.31	5	0	0	88	92.0	35	1.13	56	88.8	72	1.22	50	88.8	180	1.80	37	88.0				
.41	4	0	0	70	91.25	35	1.96	30	86.9	72	2.92	20	86.5	108	6.44	18	86.3				
Storage at 0° C.																					
.13	5	0	0	88	91.7	70	0.23	84	91.5	140	0.41	68	88.9	210	1.25	48	88.9				
.19	8	0	0	86	91.7	70	.17	106	92.0	140	.70	69	89.6	210	1.12	48	88.4				
.31	5	0	0	88	92.0	70	.74	74	90.8	140	1.32	60	87.4	210	1.53	38	87.9				
.41	4	0	0	70	91.25	70	1.44	44	88.6	140	3.39	38	87.0	210	4.69	19	86.4				
Storage at – 10° C.																					
.13	5	0	0	88	91.7	81	0.00	77	92.0	160	0.15	70	91.0	240	0.91	51	90.0	320	1.44	63	89.1
.19	8	0	0	86	91.7	81	.08	76	91.5	160	.54	58	90.6	240	.96	60	89.6	320	1.29	58	88.7
.31	5	0	0	88	92.0	81	.17	81	91.4	160	1.05	51	90.0	240	1.61	41	88.1	.....	.....	.....	.....
.41	4	0	0	70	91.25	81	.93	43	89.4	160	1.84	44	90.4	240	2.10	21	87.2	.....	.....	.....	.....
Storage at – 17° C.																					
.13	5	0	0	88	91.7	90	0.00	92	91.7	180	0.00	59	90.9	270	0.87	69	88.7	360	0.91	56	88.3
.19	8	0	0	86	91.7	90	.00	97	91.6	180	.22	65	90.8	270	.88	73	89.1	360	.96	.....	88.6
.31	5	0	0	88	92.0	90	.17	55	90.9	180	.53	55	90.2	270	1.06	66	88.4	360	1.30	49	88.0
.41	4	0	0	70	91.25	90	.68	39	88.9	180	1.58	39	89.4	270	1.70	38	87.4	360	1.88	36	87.0

difficulties encountered in the application of the dye reduction tests, the data relating to this phase of the work will not be presented here.

In order to obtain a true value for the rate of deterioration and one that would be comparable for the different butters, especially for those held at

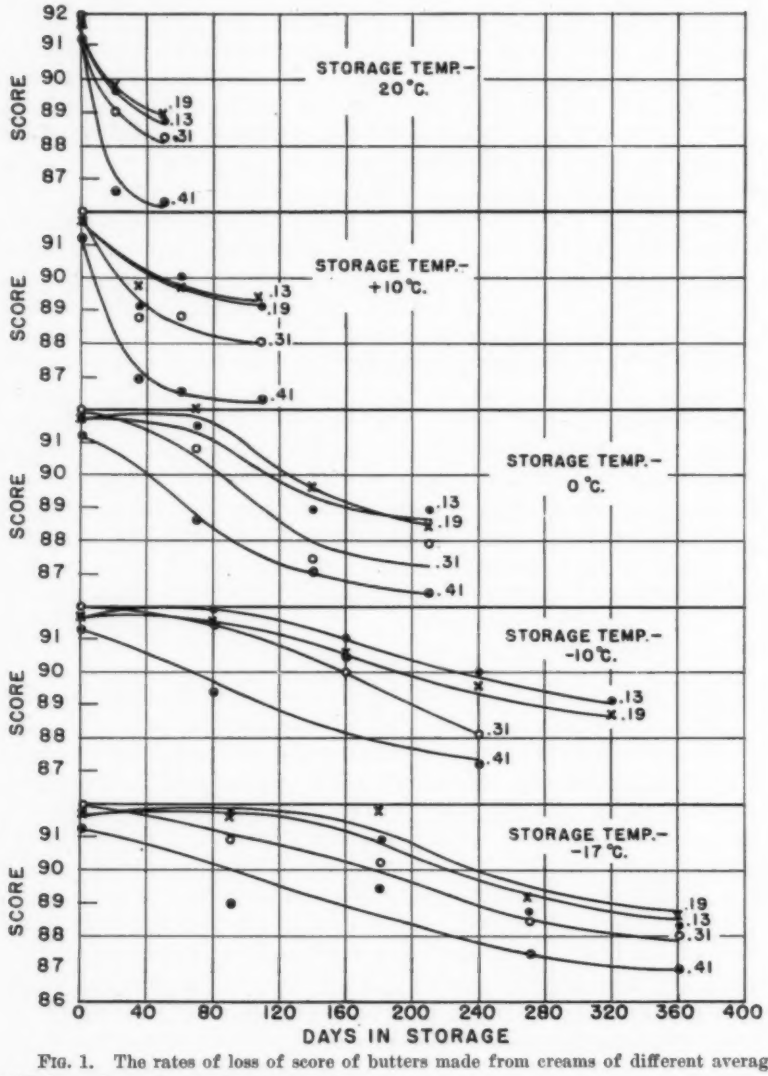

FIG. 1. The rates of loss of score of butters made from creams of different average acidities and stored at various temperatures.

TABLE 2

Scores and remarks of scorers on the various samples of butter stored for various periods of time at −17° C.

Sample No.	Acidity % Lactic	Initial		At 90 days		At 180 days		At 270 days		At 360 days	
		Score	Remarks	Score	Remarks	Score	Remarks	Score	Remarks	Score	Remarks
1–A	.13	92.0	Clean, flat	91.5	Stale	91.5	.....	90.5	.....	88.0	.....
2–A	.13	92.0	Sl. coarse	92.0	.....	91.0	.....	88.0	.....	88.0	.....
3–A	.13	92.0	.....	92.0	.....	90.0	.....	89.5	Stale, oily	.....	.....
4–A	.14	91.5	Sl. oily	90.0	.....	91.0	.....	88.0	.....	.....	.....
5–A	.12	91.0	Oily, foreign	91.5	.....	91.0	.....	87.5	.....	.....	.....
1–B	.185	91.5	.....	91.0	.....	91.0	.....	90.0	.....	89.0	.....
2–B	.18	92.0	.....	92.0	.....	90.0	.....	88.0	.....	.....	.....
3–B	.195	92.0	Clean, flat	92.5	.....	91.5	.....	88.0	.....	.....	.....
4–B	.18	92.0	.....	92.0	.....	91.0	.....	88.0	.....	.....	.....
5–B	.20	92.5	Aroma, flavor	91.5	.....	90.0	.....	89.5	Stale, oily	88.0	.....
6–B	.195	92.0	.....	92.0	.....	90.0	.....	89.5	.....	89.0	.....
7–B	.20	91.0	Over-ripe, str. flavor	91.0	Sl. oily	91.5	.....	90.0	.....	89.0	.....
8–B	.20	91.0	Oily	91.0	.....	91.0	Oily	90.0	.....	88.5	.....
1–C	.30	92.5	Starter aroma & flavor	90.0	Metallic	91.0	.....	88.0	.....	.....	.....
2–C	.36	92.0	.....	90.0	.....	90.0	.....	90.0	.....	88.0	.....
3–C	.30	92.0	Over-ripe flavor, aroma	91.+	Sl. oily	90.0	Sl. oily	89.0	Stale	88.0	.....
4–C	.30	92.0	.....	91.5	.....	91.0	.....	88.0	.....	.....	.....
5–C	.30	91.5	.....	92.0	.....	89.0	.....	87.0	.....	.....	.....
1–D	.37	91.5	Sl. oily	89.5	Metallic	91.0	.....	87.5	.....	.....	.....
2–D	.46	90.0	Oily, sl. metallic	88.0	Sl. metallic	88.0	.....	86.0	.....	.....	.....
3–D	.43	92.0	Str. aroma, sl. oily	88.0	.....	88.5	.....	87.0	Fishy	.....	.....
4–D	.40	91.5	Oily	90.0	.....	90.0	Aged	87.0	.....	87.0	.....

different temperatures of storage, the surface layer was discarded in each case before the samples to be tested were taken. After scoring, the remainder of each sample was melted at about 50° C. and the fat filtered. This fat was then used in the various tests.

The data obtained are given in the following tables and charts. The results upon each individual sample are of no particular interest, hence the averages for the samples in each particular group have been used, except in the matter of "remarks by the scorers," which of course does not lend itself to such treatment. The remarks of the scorers are given in tables 2, 3, 4 and

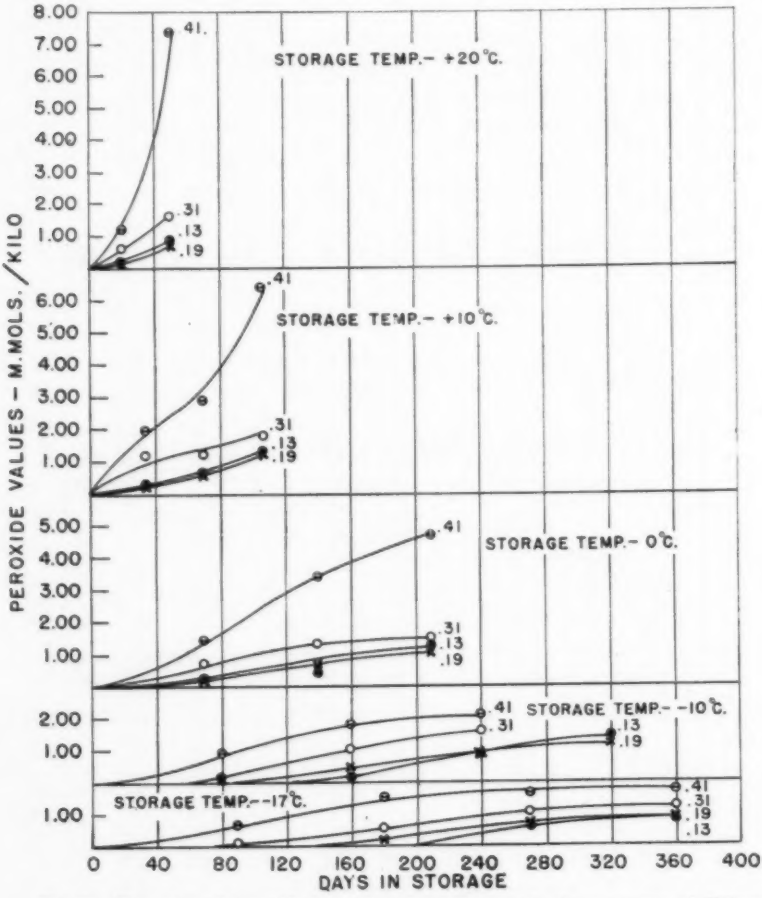

FIG. 2. The rates of peroxide formation in butters made from creams of different average acidities and stored at various temperatures.

5. Their especial significance will be discussed later. In Table 1 are given the averages of the values obtained with each test upon the samples of butter in the various groups.

Since the periods of storage between tests varied with storage temperature, it is difficult to obtain a true comparison of the rates of variation in the values from this table. Such a comparison may be made with the aid of the following graphs. The values in Figure 1 indicate the relative rate of loss in score in butters from creams of different average acidity, when the butters are stored at the indicated temperatures.

The first marked difference is that of rate of loss in score by the samples of different acidities, at each temperature of storage; except in the case of butters made from creams of 0.13 and of 0.19 per cent average acidity.

The graphs indicate further that the rates of deterioration at the lower temperatures are not constant but progress very slowly for a period of time—the length of this phase being determined by the temperature of storage—then accelerate. No "period of induction" for the changes that take place were noted in those samples stored at 10° and 20° C.

The primary stage of oxidation of fats is the addition of oxygen to the unsaturated bonds of the unsaturated acid components, to form peroxides as follows:

$$-C-C=C-C- \xrightarrow{O_2} -C-\overset{O-O}{\underset{|}{C}}-\overset{|}{C}-C-$$

These compounds are stable at ordinary temperatures in relatively low concentrations, and their amounts may be determined accurately through their ability to liberate iodine from potassium iodide in acid solution (9). Further oxidation of these compounds results in splitting of the acid with formation of compounds possessing tallowy flavors and odors. The rates of the formation of peroxides in the different butters are shown in Figure 2.

As in the case of the experiments in which the drop in score was used as a criterion of deterioration, those butters made from creams of 0.13 and 0.19 per cent average acidity show the same rate of deterioration when judged by the rate of peroxide formation. With increases in the acidity of the cream to 0.31 and 0.41 per cent, respectively, the rate of oxidation increased.

The results obtained with the bleaching test were not so consistent as those already given, but show the same order of the rates of deterioration for the different samples as was shown by the same samples of butter when judged by the loss in score or by the rate of peroxide formation.

Of the three tests discussed the peroxide value and the bleaching test relate definitely to oxidation. The score may reflect deterioration by oxidation, but is considered as a test which takes into consideration defects that may be the result of a number of different causes. Hence, to understand the type of deterioration which led to a loss in score in any particular sample,

TABLE 3

Scores and remarks of scorers on the various samples of butter stored for various periods of time at −10° C.

Sample No.	Acidity % Lactic	At 80 days		At 160 days		At 240 days		At 320 days	
		Score	Remarks	Score	Remarks	Score	Remarks	Score	Remarks
1–A	.13	92.0	.....	92.0	.....	90.5	.....	88.5	Stale
2–A	.13	92.0	.....	91.0	Oily	90.0	.....	89.0	.....
3–A	.13	92.0	.....	91.0	.....	91.0	.....	91.0	.....
4–A	.14	91.5	Very fine	90.0	.....	.....	Stale	89.0	.....
5–A	.15	91.0	Fine, clean	90.5	.....	89.0	Stale	88.0	.....
1–B	.185	91.5	.....	91.0	Sl. oily	90.0	Stale, oily	89.0	Stale
2–B	.18	91.5	Clean	90.0	.....	89.0	Stale	87.0	.....
3–B	.195	92.0	.....	91.5	Sl. oily	90.0	.....	88.5	.....
4–B	.18	92.0	.....	91.0	Oily	90.0	.....	89.0	.....
5–B	.20	92.0	.....	91.0	.....	91.0	.....	90.5	.....
6–B	.195	91.5	.....	91.0	.....	90.0	Stale	88.0	Stale, oily
7–B	.20	91.0	.....	90.5	.....	88.0	Stale, oily	.....	.....
8–B	.20	91.0	.....	89.0	.....	89.0	.....	89.0	.....
1–C	.30	91.0	.....	91.0	.....	88.0	.....	.....	.....
2–C	.36	91.0	.....	91.0	.....	88.0	Stale	.....	.....
3–C	.30	91.0	.....	88.0	Cooked, curdy, old	88.0	Fishy	.....	.....
4–C	.30	92.0	.....	90.0	.....	.....	Stale	.....	.....
5–C	.30	92.0	Fine	90.0	Sl. aged	88.5	Stale, oily	.....	.....
1–D	.37	90.0	Sl. metallic	91.0	Oily	88.0	.....	.....	.....
2–D	.46	88.0	Metallic	90.0	Stale, oily	87.0	.....	.....	.....
3–D	.43	88.0	Metallic	90.0	Old, oily	87.0	Fishy	.....	.....
4–D	.40	91.5	Metalic	90.5	Aged	87.0	Fishy	.....	.....

TABLE 4.

Scores and remarks of scorers on the various samples of butter stored for various periods of time at 0° C.

Sample No.	Acidity % Lactic	At 70 days		At 140 days		At 210 days	
		Score	Remarks	Score	Remarks	Score	Remarks
1–A	.13	91.0	Stale	89.5	Aged	88.0	.....
2–A	.13	92.0	.....	89.0	Sl. oily	88.0	.....
3–A	.13	92.0	.....	89.0	Storage flavor	90.0	.....
4–A	.14	90.5	Sl. coarse	88.5	.....	89.5	Stale
5–A	.12	92.0	Sl. coarse salt	88.5	.....	.....	Stale, oily
1–B	.185	92.0	.....	91.0	Coarse	88.0	Stale, oily
2–B	.18	92.0	.....	87.0	Cheesy	87.0	.....
3–B	.195	92.0	.....	89.0	Oily, sl. bitter	88.5	.....
4–B	.18	92.0	.....	88.0	Oily, sl. bitter	88.5	.....
5–B	.20	92.0	.....	89.0	Oily	90.5	.....
6–B	.195	92.0	.....	91.0	.....	88.0	Stale, oily
7–B	.20	92.0	.....	90.0	Storage & sl. acid	89.0	.....
8–B	.20	92.0	.....	92.0	.....	88.0	.....
1–C	.30	91.0	Stale	87.0	Fishy, greasy	88.0	.....
2–C	.36	91.0	Starter flavor	87.5	Fishy	87.5	Stale, oily, sl. metallic
3–C	.30	91.5	.....	88.0	Storage, sl. acid	88.0	.....
4–C	.30	89.0	S. metallic, soapy	88.5	.....	89.0	Stale
5–C	.30	91.5	.....	87.0	Cheesy	87.0	.....
1–D	.37	90.0	S. metallic	88.0	Oily, sl. metallic	87.0	.....
2–D	.46	87.0	Fishy	85.0	Bitter, old, lardy, rancid	84.0	Bleached
3–D	.43	87.0	Fishy	87.0	Old, rancid	87.0	Fishy
4–D	.40	90.0	Sl. metallic	87.0	Fishy	87.0	Stale, oily, sl. metallic

TABLE 5

Scores and remarks of scorers on the various samples of butter stored for varying periods of time at 10 C.

Sample No.	Acidity % Lactic	At 35 days		At 72 days		At 108 days		At 140 days	
		Score	Remarks	Score	Remarks	Score	Remarks	Score	Remarks
1–A	.13	88.5	Cheesy	89.0	Stale, musty	87.0	Moldy	.....	.....
2–A	.13	89.0	Stale	89.0	Stale, musty	89.0	.....	.....	.....
3–A	.13	90.0	Storage, oily	90.0	Stale	90.0	Stale	89.0	Stale
4–A	.14	.....	.....	91.0	.....	90.5	.....	90.0	.....
5–A	.12	.....	.....	90.0	.....	89.0	Stale	.....	.....
1–B	.185	90.0	Storage	90.0	Stale	90.0	Stale	89.0	Stale
2–B	.18	.....	.....	89.5	Musty	89.5	Cheesy	89.5	Stale
3–B	.195	88.0	Sl. cheesy, old	89.0	Stale, musty	88.0	Stale, sl. moldy	.....	.....
4–B	.18	90.0	.....	89.0	Stale, musty	89.0	.....	.....	.....
5–B	.20	90.5	Old, coarse	89.0	Stale	89.0	Stale	.....	.....
6–B	.195	90.0	Old, oily	90.0	"	90.0	Stale	89.0	Stale
7–B	.20	90.0	Sl. oily	90.0	"	90.0	.....	89.0	Stale
8–B	.20	90.0	Oily	90.0	"	89.5	.....	.....	.....
1–C	.30	89.0	Stale, sl. metallic	88.5	Tallowy	87.0	Fishy	.....	.....
2–C	.36	89.0	Sl. fishy	88.5	Metallic	88.0	Stale, oily	.....	.....
3–C	.30	88.5	Old, oily	89.0	Stale	88.0	Stale, oily	.....	.....
4–C	.30	.....	.....	90.0	Stale	90.0	.....	89.5	.....
5–C	.30	.....	.....	88.0	Stale, oily	88.0	Cheesy, stale, oily	.....	.....
1–D	.37	88.0	Stale, oily	87.0	Metallic	86.0	Fishy	.....	.....
2–D	.46	85.0	Fishy, bleached	85.0	Fishy	85.0	Bleached, stale, oily	.....	.....
3–D	.43	87.0	Fishy	86.0	Bleached, fishy	86.0	Bleached, stale, oily	.....	.....
4–D	.40	87.5	Stale	88.0	Metallic	88.0	Stale, oily	.....	.....

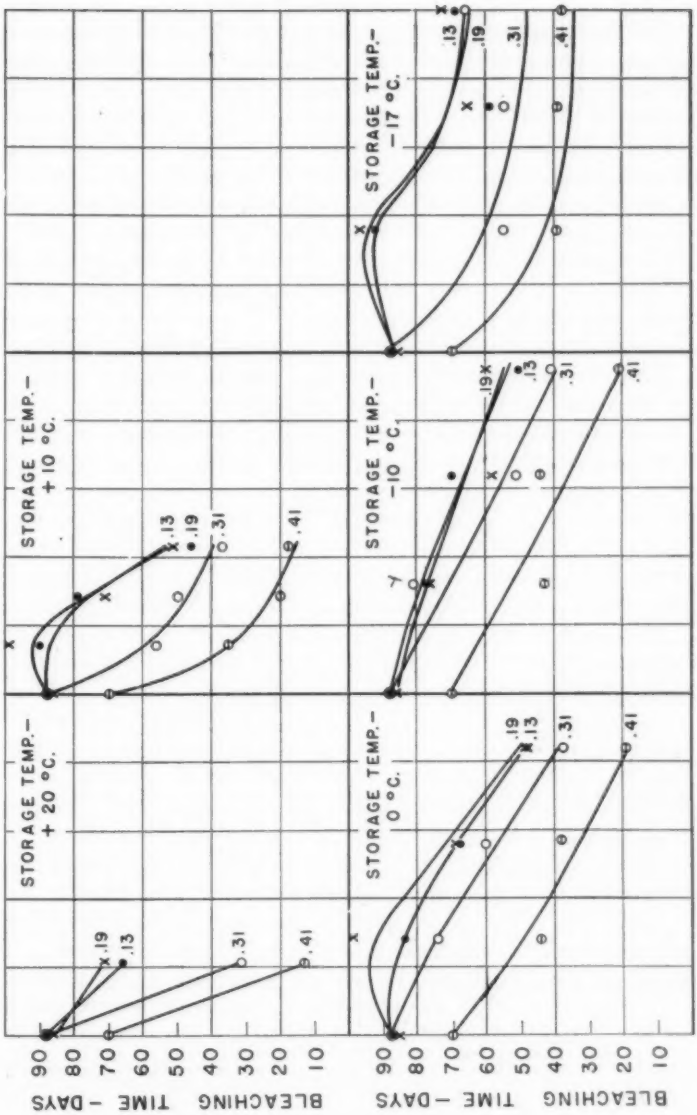

FIG. 3. The time necessary at 42° C. for bleaching of butteroils from butters made from creams of different average acidities and stored at various temperatures.

the remarks of the scorers are of especial value. These are given in Tables 2, 3, 4, and 5.

A survey of these tables indicates little or no difference between the butters made from creams of 0.13 and 0.19 per cent acidity in the type of deterioration during storage. In none of these samples did the scorers note a metallic, tallowy, or fishy flavor, although some samples were stored for 360 days at −17° C. Samples of butter made from 0.30 and 0.40 per cent acid cream possessed metallic flavors and fishy flavors relatively early in the storage period, the time of their occurrence depending upon the temperature of storage and the acidity of the creams from which they were made. Metallic and fishy flavors occurred more frequently and earlier in the storage period in the butters made from creams of 0.40 per cent acidity than in that made from 0.30 per cent acidity.

The relative efficiency of the different temperatures in promoting keeping quality of the different butters may be determined from the values in Table 1. The relative efficiencies have been shown graphically by plotting the time of storage in days required by each butter at each storage temperature, to result in a loss of two points in score.

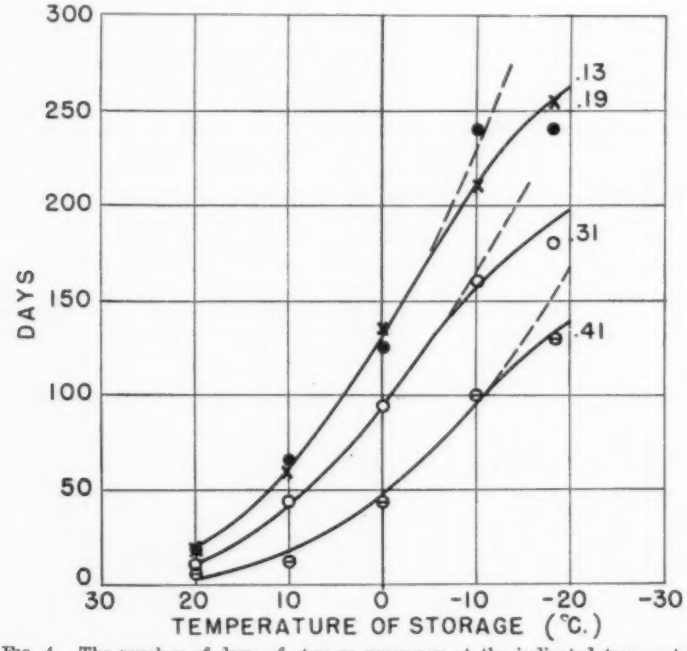

FIG. 4. The number of days of storage necessary at the indicated temperatures to result in a loss of two points in the score of butters made from creams of different acidities.

A decrease of the temperature from – 10° C. to – 17° C. does not seem to decrease the susceptibility of butters to deterioration to so great an extent as do similar decreases in the higher temperature ranges.

The graphs indicate also in a general manner the relative keeping quality of butters made from creams of different acidities.

DISCUSSION

Butters made from creams of different acidities grouped themselves similarly when their keeping quality was judged by any of the three tests used. In each case practically no difference was noted in the values obtained upon butters from creams of 0.13 and 0.19 per cent acidity at any of the chosen temperatures of storage. In other words, the rates of deterioration of sweet cream butters and of butters made from creams of less than 0.20 per cent acidity, developed by pure cultures added to sweet creams, were practically identical. Whether or not butters made from creams of acidities slightly greater than 0.20 per cent, possess a similar rate is not indicated by the data. Butters made from cream of an acidity of 0.30 per cent or more were found to be inferior in keeping quality to those made from sweet cream.

From the standpoint of reliability in judging deterioration, the score method will probably always be preferred, because of its direct relationship to edibility. However, it may not be directly related to any one type of chemical change and lacks in exact quantitative aspects, especially after the initial deterioration changes have taken place. Below a score of 89 the drop in score does not seem to correlate directly with the magnitude of the chemical changes that take place. This is noted especially in the relationship between scores and peroxide values of the samples stored at 20°, 10° and 0° (see Figs. 1 and 2).

The general similarity in the rates of deterioration, determined by the loss in score and by the development of peroxide, throughout the period of storage, in spite of the fact that in scoring all off flavors and odors are taken into consideration, seems to verify the hypothesis that there is a direct relationship between rate of oxidation and loss in score, or stated more directly, the oxidation reaction seems to underlie the various changes that are responsible for the loss in score.

A measure of the rate of oxidation of a butterfat seems, therefore, to be a direct measure of the rate of deterioration even though the direct end product of this reaction—tallowiness—may not be noted in the scoring.

Fishy flavors and odors in butter have been shown to be caused by an acid medium and especially when accompanied by oxidation promoted by metals (3) (10). The present results verify the conclusion that acids promote this type of deterioration and show also that spontaneous autoxidation will aid its development. However, it appears that an acid medium is the major factor concerned, for the amount of peroxides developed in sweet cream butters was

often equal to or greater at the end of the storage period than the amounts in the acid cream butters when the latter possessed a fishy flavor, yet none of the butters made from cream of an acidity of 0.20 per cent or less developed a fishy flavor. The exact rôle that oxidation may play in the development of fishy flavors is not clear.

The efficiency of different temperatures of storage in promoting keeping quality is shown in Figures 1, 2, and 3. Their relative value is shown in Fig. 4, which seems to indicate that a drop in storage temperature from – 10° C. to – 17° C. does not result in an increase in keeping quality proportionate to that observed for a similar lowering at a higher temperature range.

ACKNOWLEDGMENT

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SPECIFIC HEAT AND THE PHYSICAL STATE OF THE FAT IN CREAM

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INTRODUCTION

The physical state and alterations in the physical state of the fat in the globules of milk and cream offer the most reasonable explanation for the profound effect of temperature on creaming, cream viscosity, foaming, churning, lipase activity, and surface tension. Many of these effects are reversible and are probably produced or influenced by the materials adsorbed on the surface of the globules when the fat is in different physical states. In order better to understand the influence of temperature on milk products, a knowledge of the physical state and alterations in the physical state of the fat in the globules is necessary. Information obtained by a study of milkfat in mass cannot be used as indicating accurately the physical state of the fat dispersed as globules. Seeding occurring in a mass of fat will influence the crystallization of the whole mass, but in fat dispersed as globules seeding will affect only the fat in the globule in which the crystal happens to form, and will exert no direct effect on the fat in other globules. This leads to a much slower attainment of equilibrium. Furthermore the crystals are larger in fat in mass, as compared with fat in globules, and this influences the rate of solution or melting on heating. Crystallization in both fat in mass and in globules is influenced by the rate of cooling.

A study was undertaken to gain information as to the physical state of the fat in globules at different temperatures and the rate of attainment of a constant physical state at the different temperatures. Determinations of specific heat were used as one method of following the alterations in the state of the fat in the fat globules.

Chevreul (3) observed that the solidification of milkfat was accompanied by the evolution of heat. Fleischmann (5) (6), Fjord (4), Landois (9), Chanoz and Vaillant (2), Schnorf (10), Hammer and Johnson (7), and Bowen (1) have reported specific heat values for milk and cream. The effect of fat has been studied more specifically by Fleischmann (6), Hammer and Johnson (7), and Bowen (1), the latter reporting values obtained by the U. S. Bureau of Standards. There is no assurance that the fat was in equilibrium at the different temperatures used. The use of an ordinary calorimeter involves difficulties when used to determine the specific heat of milk and cream at temperatures within crystallization range, because considerable time is required to attain the equilibrium state, and during this

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time the heat exchange between the calorimeter and its surroundings makes up an ever increasing part of the total heat measured.

EXPERIMENTAL

Method

The method of mixtures was used, since it avoids the errors due to large values for heat exchange with the surroundings, and has a reputation for high accuracy. Quart thermos bottles were used as containers. Warm water from one bottle was poured into colder cream or milk contained in another. The thermometers used were graduated to 0.1° C. and were calibrated against each other over the range used—from 0 to 75° C. All readings were made with the aid of a magnifying glass and were estimated to 0.02° C. The errors and heat losses in the procedure were studied, and all equipment and procedures were thoroughly standardized. The heat "loss" to the container and during the manipulation was found to be given by the following equation:

$$\text{Loss calories} = K_1(t_f - t_i) + K_2(t_w - t_r)$$

where K_1 and K_2 are constants representing the heat equivalent of the apparatus and heat lost in pouring, respectively,

t_f = final temperature of cream-water mixture,

t_i = initial temperature of the cream,

t_w = the temperature of the warm water before pouring,

and t_r = the temperature of the room.

It was found by experiment that within the normal limits, the relative humidity of the room exerted an effect so small that it could be disregarded. The constants were determined by calibration with water, the room temperatures varying from 23 to 28° C., cold water varying from 5 to 39° C., and warm water varying from 21 to 66° C. The constants were calculated by the method of least squares, and for a typical pair of thermos bottles gave the equation:

$$\text{Loss calories} = 25.35\ (t_f - t_i) + 3.38\ (t_w - t_r)$$

About 150 grams of cream and 250 grams of water were used. The amounts were determined by weighing. The specific heat of water was taken as unity, and after correcting for the heat loss, the specific heat of the cream was calculated.

Results

The specific heat of skimmilk was determined both by the method of mixtures and by means of the conventional calorimeter in which heat was produced by an electric heater immersed in the milk. Within rather narrow limits the same values were obtained by both methods. This indicates that the heat of dilution involved in the method of mixtures is negligible. The average value was found to be 0.943 with only slight deviations for the

various samples over the temperature range studied. The specific heat of the fat in the cream was calculated from the specific heat of the skimmilk, the specific heat and the fat content of the cream.

Cream of about 40 per cent fat content obtained from mixed milk was first warmed to 45° C. and was then cooled rapidly by immersing the container in water the temperature of which was about 5° C. below the final temperature desired for the cream. As soon as the desired temperature of the cream was reached, the temperature of the water bath was adjusted to the temperature of the cream, and an aliquot of the cream was removed at once and its specific heat determined. Determinations of specific heat were continued at intervals until it was evident that a constant value for that particular temperature had been reached. Before introducing the cream into the bottle, the temperature of the thermos bottle was adjusted to that of the cream by means of water. The temperature of the warm water which was poured into the cream was so adjusted that the resulting mixture would

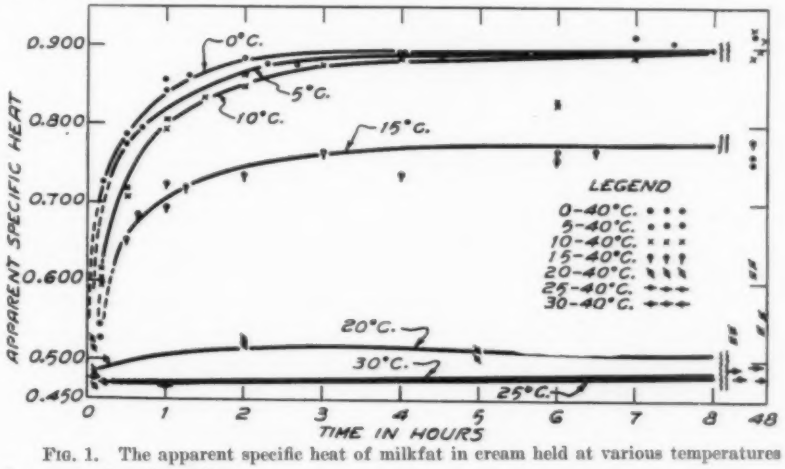

FIG. 1. The apparent specific heat of milkfat in cream held at various temperatures for varying lengths of time.

have a temperature of 40° C. or slightly above. In this way values were obtained for the heat required to warm to 40° C. cream having original temperatures ranging from 0 to 30° C. The specific heat values thus obtained are the average specific heats for the temperature ranges. The specific heat values attributable to the fat alone are presented graphically in Figure 1. This figure indicates that if the cream is cooled rapidly enough the fat at rather low temperatures may for a short time still be liquid and have the specific heat of liquid fat. Crystallization soon begins, however, and continues for about 4 hours, since the specific heat increases for a period of approximately 4 hours. After this time the change is slight.

The effect of season and feed of the cow on the hardness of the fat has been studied by Hunziker, Spitzer and Mills (8) and others. The fat is generally less hard on pasture than on dry feeding. Figure 2 shows that there

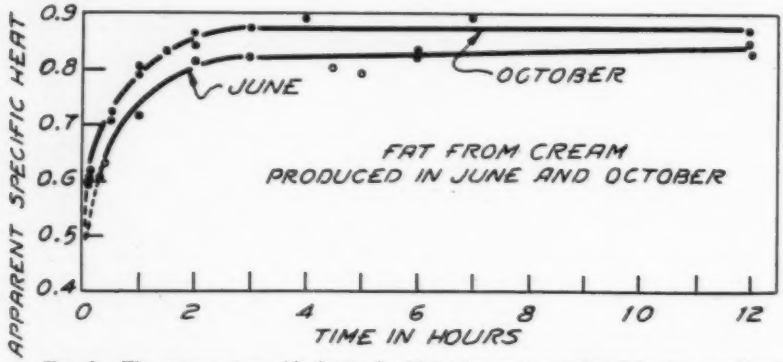

FIG. 2. The apparent specific heat of milkfat in cream produced in the month of June and in October. Cream warmed from 10° to 40° C.

is also a difference in specific heat of the fat in the temperature range in which crystallization of fat occurs.

Aliquots of cream, one at equilibrium at 15° C., the other at equilibrium at 20° C., differ in the number of calories required to warm the cream from equilibrium at 15 to equilibrium at 20° C. by the difference in the amount of heat required to raise each to 40° C., a temperature at which the fat is liquid. Using this procedure, the heat required to raise milkfat from successive equilibrium states at 5° C. intervals in temperature was calculated. In Table I are recorded the average equilibrium specific heat values of the fat calculated

TABLE 1

Apparent specific heat of fat in cream for each 5° C. interval ranging from 0 to 40° C. Milk produced in October and November

Temp. range	Temperature difference	Apparent sp. heat	Heat required	Heat required 5° C. interval	Average sp. heat for interval	Temperature interval
°C.	°C.		*calories*	*calories*		°C.
					0.475	30–40
30–40	10	0.475	4.75			
				2.38	0.475	25–30
25–40	15	0.475	7.13			
				2.87	0.575	20–25
20–40	20	0.50	10.00			
				9.25	1.85	15–20
15–40	25	0.77	19.25			
				7.15	1.43	10–15
10–40	30	0.88	26.40			
				4.75	0.95	5–10
5–40	35	0.89	31.15			
				4.45	0.89	0– 5
0–40	40	0.89	35.60			

from the calories required to warm to 40° C. cream held at a series of temperatures. The total number of calories required to warm the fat through the given temperature range was then calculated, and by difference the increments in calories for each 5 degrees. In the next to the last column the average specific heat of the fat in each 5 degree range is given. This table indicates that the apparent specific heat of the fat varies from 0.475 for liquid fat to 1.85 for the temperature range between 15 and 20° C., the range in which the greatest change in physical state occurs. If smaller increments in the region of 15° C. had been taken, still higher values might be obtained. The data in this table are in general agreement with those of Fleischmann (6), Hammer and Johnson (7), and Bowen (1). The data from Table 1 were used to calculate the specific heat increments over the temperature range of from 0 to

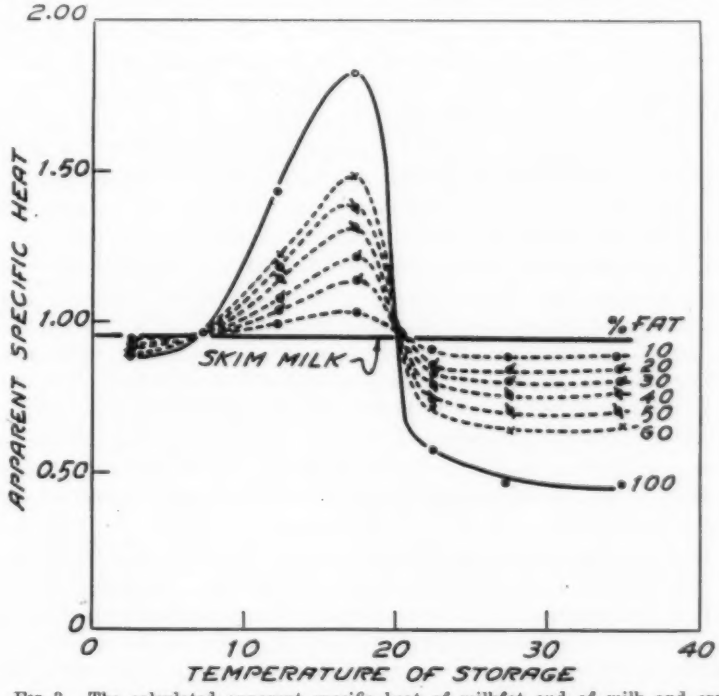

FIG 3. The calculated apparent specific heat of milkfat and of milk and cream, within each 5° C. interval in the 0–30° C. and in the 30–40° C. range. The calculations are based on values obtained on samples which had been stored at the various temperatures for 4 hours.

40° C. of milk and cream containing various percentages of fat, and the results are given in Table 2. The relationships are more readily visualized in Figure 3.

TABLE 2

Specific heat of skimmilk, cream, and milk fat globules for 5° intervals ranging from 0 to 40° C. milk produced in October and November

Temperature interval °C.	Fat content per cent								
	0	5	10	20	30	40	50	60	100
0– 5	.943	.940	.937	.932	.927	.921	.916	.911	.890
5–10	.943	.943	.943	.944	.945	.945	.946	.947	.950
10–15	.943	.967	.991	1.040	1.089	1.138	1.186	1.235	1.43
15–20	.943	.988	1.033	1.124	1.215	1.305	1.396	1.487	1.85
20–25	.943	.924	.905	.869	.832	.795	.758	.722	.575
25–30	.943	.919	.895	.849	.802	.755	.708	.662	.475
30–40	.943	.919	.895	.849	.802	.755	.708	.662	.475

DISCUSSION

The following melting point temperatures are typical of those given in the literature: tristearin 71° C., tripalmitin 65° C., trimyristin 55° C., and triolein –5° C. Stearic, palmitic, myristic and oleic are the principal acid components of milkfat. Milkfat may be considered as a solution which on cooling becomes supersaturated with respect to one or more of the mixed glycerides of which it is composed. The uncertainty and indistinctness of the so-called solidifying point would be expected, since a true solidifying point is not involved. The temperature at which a supersaturated solution begins to crystallize varies with the conditions, such as degree of supersaturation, agitation, seeding, etc. Furthermore, after crystallization starts the rate of crystallization of a solute from its supersaturated solution may vary greatly and crystallization may extend over a considerable period of time. The crystallization of milkfat is still further complicated because on cooling it probably becomes supersaturated with respect to more than one solute. Milkfat can readily be fractionated into one component which will not melt unless heated above 45° C., and into another which will not solidify when cooled to 5° C.

The so-called melting point of milkfat is not definite because we are really dealing with a temperature-solubility relationship. As the milkfat is warmed, the solid glycerides become more soluble in the liquid fractions, and an appreciable increase in solubility is noticed in the region of 15° C., but complete solution is usually not obtained until the fat is heated above 30° C. The rate at which the crystals dissolve is dependent upon their size, which in turn is dependent upon the rate and conditions of the previous cooling. These phenomena are all demonstrable with milkfat in mass.

On cooling, the lag in the formation of the solid phase in milkfat in the globule state is much greater than for fat in mass.

The alteration of the specific heat of cream with time was used as a method

for following the rate of formation of solid milkfat at the various temperatures. Below 20° C., the greater part of the crystallization is complete in about four hours. This is in agreement with some experiments carried out by Troy and Sharp (11), who showed that solidification of the fat globules, as indicated by resistance to pressure in the centrifuge, was not complete in two hours, but was complete in five hours, at 3° C.

CONCLUSIONS

1. The method of mixtures is a reliable method for the determination of specific heat of cream within the range in which a change in the relative amounts of crystalline and liquid fat occurs.

2. When cream is cooled to temperatures within the range of 0 to 20° C., the phase adjustment is nearly complete in about 4 hours, so far as this is indicated by the specific heat.

3. The average equilibrium specific heat for milk-fat in the globules for 5° C. temperature intervals is as follows: 0–5° C., 0.89; 5–10° C., 0.95; 10–15° C., 1.43; 15–20° C., 1.85; 20–25° C., 0.575; 25–40° C., 0.475; for fat in milk produced in October and November.

4. A variation in the specific heat of the milkfat with feed or season was shown.

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BOUND WATER AND ITS RELATION TO SOME DAIRY PRODUCTS.

II. FACTORS AFFECTING THE BOUND WATER CONTENT OF SOME DAIRY PRODUCTS*

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Evidence that liquid dairy products contain bound water was presented in the first of this series of studies on bound water (1). There was also some indication that the bound $\rightleftharpoons$ free water equilibrium could be changed readily by various treatments. Sayre (2) states that "Bound water does not exist in definite proportions relative to the solid material of the system, but as a ratio between bound water and free water. The ratio may be changed quickly by varying the temperature, acidity, surface energy, presence of electrolytes, pressure, etc."

Since the degree of hydration plays a rôle in giving colloidal stability to the product in question, it was thought advisable to determine some of the factors that influence the bound water content. If the product in question lacks colloidal stability, during or after manufacture, in many cases a partial precipitation of the proteins may occur. It can be readily seen that the nature of some of the defects that occur in ice cream, milk, evaporated milk, sterilized cream, etc., are usually associated with the ability of the milk proteins to bind water or to readsorb water after certain necessary treatments.

It has been known for a long time that certain effects are produced by various treatments of dairy products but just what causes some of these effects has never been definitely established. Therefore, a study of the factors affecting the bound water content of some dairy products was undertaken in the hope that some of these phenomena might be explained more fully.

EXPERIMENTAL METHODS

The samples used, unless otherwise stated, were obtained from the same sources as previously mentioned (1). The experimental methods used were described before (1).

The data on aging, viscosity and protein stability given throughout this work will be dealt with in detail in the last paper of this series.

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** The data presented in this paper are from a thesis submitted to the Graduate School of The Pennsylvania State College in partial fulfillment of the degree of Doctor of Philosophy, 1937.

The Effect of pH on the Bound Water Content of Casein Sols

A study was made of the effect of pH on the bound water content of casein sols. The casein used was pure casein (Pfanstiehl). It was dispersed with sodium bicarbonate to obtain the desired pH values. The effect of increased and decreased pH was determined, using a casein sol at a pH of 6.69 as the control. Table 1 gives a summary of the data obtained on the effect of pH on the bound water content of casein sols.

TABLE 1

The effect of pH on the bound water content of casein sols

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Per cent bound water	Grams bound water per gram solids
1. Casein Sol (pH 5.83)	4	2.79	5.81	1.244	0.125	1.06	0.38
	24	2.79	5.83	1.283	0.125	1.24	0.44
2. Casein Sol (pH 6.69)	4	2.97	6.64	1.679	0.12	2.29	0.77
	24	2.97	6.69	1.718	0.12	2.42	0.81
3. Casein Sol (pH 7.36)	4	3.51	7.34	1.442	0.115	1.37	0.39
	24	3.51	7.36	1.540	0.115	1.77	0.50

The hydrogen ion concentration is probably one of the most important factors involved in the study of the bound water content of dairy products. It probably has a definite influence on the amount of water bound in dairy products since most proteins have well defined isoelectric points or zones.

The studies with casein sols in Table 1 show the importance of optimum pH in order to have maximum bound water content. Increasing or decreasing the pH from the "normal" brings about a decrease in the bound water content. A decrease in pH appears to be slightly more detrimental to the bound water content than an increase in pH.

The Effect of pH on the Bound Water Content of Concentrated Milk Plasma

The pH of normal ice cream mixes made from fresh products is usually between 6.2 and 6.4. Since pH plays such an important part in protein stability, it was thought that altering the pH of a concentrated milk plasma would affect the bound water content. Sodium bicarbonate was used to change the pH in the two samples. The pH of the fresh control was 6.22 which is normal for an ice cream mix. The results of these trials appear in Table 2.

Increasing the pH in the range studied has a marked effect on the water binding capacity of the concentrated milk plasma as can be seen from Table 2. A decrease in the bound water content resulted when the pH was increased to neutrality and slight alkalinity. It will be noted that although

TABLE 2

The effect of increasing the pH on the bound water content of concentrated milk plasma

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water	Grams bound water per gram solids
1. Conc. Milk Plasma pH 6.22	0	17.57	6.22	5.590	0.38	7.2	6.10	0.347
	4	17.57	6.24	6.183	0.375	7.3	6.76	0.384
	24	17.57	6.22	7.051	0.375	7.4	8.12	0.462
2. Conc. Milk Plasma pH 7.05	0	17.01	7.05	6.183	.....	9.1	4.78	0.281
	4	17.01	7.08	5.293	.....	8.9	5.60	0.323
	24	17.01	7.08	6.459	.....	9.2	6.62	0.383
3. Conc. Milk Plasma pH 7.35	0	17.31	7.35	6.183	.....	9.4	3.86	0.223
	4	17.31	7.37	6.755	.....	9.2	4.77	0.275
	24	17.31	7.37	6.459	.....	9.5	4.05	0.234

a decrease in bound water content resulted on increasing the pH, the protein stability as measured by the alcohol number increased.

Another series of samples was made and the effect of decreasing the pH on concentrated milk plasma was determined. Lactic acid was used to lower the pH of the samples.

The pH was decreased to 5.97 and 5.19 while the pH of the control was 6.31. The results of these mixes are recorded in Table 3.

TABLE 3

The effect of decreasing the pH on the bound water content of concentrated milk plasma

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water	Grams bound water per gram solids
1. Conc. Milk Plasma pH 6.31	0	14.75	6.31	5.293	0.300	7.8	2.37	0.161
	4	14.75	6.32	5.590	0.290	7.8	3.69	0.250
	24	14.75	6.32	5.886	0.290	7.7	5.37	0.364
2. Conc. Milk Plasma pH 5.97	0	15.14	5.97	4.701	0.575	3.0	0.31	0.020
	4	15.15	6.05	4.997	0.560	3.0	1.76	0.116
	24	15.14	6.05	5.590	0.560	3.0	3.08	0.203
3. Conc. Milk Plasma pH 5.19	0	15.14	5.19	4.701	0.590	2.0	0.11	0.007
	4	15.14	5.23	4.997	0.590	1.6	2.17	0.143
	24	15.14	5.23	4.997	0.595	1.5	1.72	0.113

It is very evident that a decrease in pH in the range studied decreases the bound water content of concentrated milk plasma. It is logical to believe that further decreases in pH toward the isoelectric zone would decrease the bound water content still more and that it should be at a minimum at the isoelectric point. It appears then that the phenomenon of precipitation is accompanied by a decrease in the bound water content.

It will be noted also that with the decrease in bound water content there was a decrease in viscosity and protein stability.

The Effect of Concentration on the Bound Water Content of Casein Sols

The casein used for this study was pure casein (Pfanstiel). It dispersed readily when the pH was adjusted with sodium bicarbonate. Sodium caseinate was undoubtedly formed which is more soluble than calcium caseinate and probably binds more water, but the results nevertheless indicate the effect of concentration on the bound water content of these casein sols. These results are recorded in Table 4.

TABLE 4

The effect of concentration on the bound water content of casein sols

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Per cent bound water	Grams bound water per gram solids
1. Casein Sol (1.67%)	4	1.67	6.98	1.185	0.07	1.46	0.87
	24	1.67	6.97	1.244	0.07	1.53	0.92
2. Casein Sol (3.35%)	4	3.35	6.91	1.718	0.11	2.33	0.70
	24	3.35	6.89	1.758	0.11	2.50	0.75
3. Casein Sol (6.71%)	4	6.71	6.83	4.345	0.24	3.54	0.53
	24	6.71	6.81	4.444	0.24	4.42	0.66

The bound water content per gram of casein tends to diminish with the concentration of the casein sol. In general, it is known that a hydrophilic colloid will bind more water per gram of substance in dilute solution. Casein, then, acts like a hydrophilic colloid in this respect and seems to show that bound water is closely associated with concentration. Chrysler (3), working with kelp, has found similar results. Newton and Martin (4), experimenting on some colloidal sols, show that in most cases the water bound per gram of colloid tends to diminish with the concentration of the sols.

The Effect of Varying Temperatures on the Bound Water Content of Concentrated Milk Plasma

It is well known that the temperature of pasteurization of dairy products affects the physico-chemical properties of the product. It seems probable that varying temperatures would affect the bound water content. Certain high heat treatment is known to favor protein stability with alcohol but what effect it has on hydration has not been definitely known.

Three samples of concentrated milk plasma were compared. The control was heated to 145° F. for 30 minutes while another sample was heated to 175° F. for 5 minutes and the third sample was boiled for one minute. The data for this experiment are given in Table 5.

It will be seen in Table 5 that the higher heat treatment reduced the bound water content of the concentrated milk plasma. It would seem natural to expect that at high temperatures the colloidal micelles are partially

TABLE 5

The effect of heat treatment on the bound water content of concentrated milk plasma

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water	Grams bound water per gram solids
1. Heated 145° F. for 30 min.	0	13.80	6.38	3.970	0.29	7.9	4.01	0.291
	4	13.80	6.38	4.108	0.285	8.0	4.73	0.343
	24	13.98	6.38	3.239	0.285	8.0	2.56	0.183
2. Heated 175° F. for 5 min.	0	13.98	6.31	3.516	0.285	8.0	3.42	0.245
	4	13.98	6.36	3.832	0.28	8.1	4.41	0.315
	24	13.98	6.38	3.239	0.285	8.0	2.56	0.183
3. Boiled one minute	0	14.29	6.27	3.970	0.29	8.2	2.51	0.176
	4	14.29	6.33	4.108	0.29	8.3	3.28	0.230
	24	14.29	6.34	3.970	0.29	8.2	2.71	0.190

dehydrated and therefore a decrease in bound water content results. At the higher temperatures it will be seen that the protein stability as measured by the alcohol number increased but slightly. It will also be noted that the bound water content was lower after 24 hours of aging at 40° F. than after 4 hours of aging.

The Effect of Heat on the Bound Water Content of the Fat Globule "Membrane" and Pure Milk Phospholipids

Perlman (5) and Jack (6) have shown the effect of heat on the milk phospholipids. The latter worker believes that the action of heat is a denaturation process in removing the phospholipids from the surface of the fat globules. The writers have further observed that when cream containing approximately 50 per cent of butterfat is heated to 160° F. or higher, some "oiling off" of the fat will result if the cream is not stirred.

Fat globule "membrane" was prepared from washed cream as previously mentioned (1). To note the effect of heat on the bound water content of this material, samples were prepared and heated to various temperatures and aged 4 and 24 hours at 40° F. The samples used were: (1) unheated

TABLE 6

The effect of heat on the bound water content of the fat globule "membrane"

Sample	Hours aged at 40° F.	Per cent solids	pH	Per cent bound water	Grams bound water per gram solids
1. Unheated	4	2.52	6.32	1.55	0.615
	24	2.52	6.05	1.77	0.702
2. Heated to 143° F. for 30 min.	4	2.42	6.53	1.38	0.570
	24	2.42	6.43	1.50	0.619
3. Heated to 160° F. for 5 min.	4	2.49	6.63	0.90	0.361
	24	2.49	6.58	1.06	0.426

control; (2) heated to 143° F. for 30 minutes; and (3) heated to 160° F. for 5 minutes. The bound water studies and other observations are given in Table 6.

The effect of heat on the fat globule membrane is similar to that obtained for other fluid dairy products. A slight decrease of bound water content was obtained when the membrane material was heated to 143° F. for 30 minutes. A temperature of 160° F. for 5 minutes decreased the bound water content markedly.

Some observations on the appearance and odor of the material on heating indicate that some chemical change took place. The unheated control had the appearance of rich milk, had a creamy consistency and gave off no odor, while the sample heated to 143° F. for 30 minutes was lacking somewhat in creamy appearance and gave off a slight sulfur odor. The sample heated to 160° F. for 5 minutes was completely bleached in color, had a watery consistency and gave off a strong odor of sulfur similar to hydrogen sulfide.

An increase in pH and a decrease in acidity occurred on heating the fat globule "membrane." Possibly this reduction in acidity is due to the liberation of carbon dioxide. The viscosity studies show that there was a noticeable decrease on heating. This may have some bearing on the decrease in viscosity of cream on pasteurization.

The effect of heat on the bound water content of pure milk phospholipids was also studied. The pure milk phospholipids material was made as previously mentioned (1). The same temperatures of heating were used as in the previous experiment on the fat globule "membrane." Table 7 shows the results obtained in this experiment.

TABLE 7

The effect of heat on the bound water content of pure milk phospholipids

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Per cent bound water	Grams bound water per gram solids
1. Unheated	4	0.66	6.83	2.163	0.005	3.53	5.35
	24	0.66	6.82	2.204	0.005	3.95	5.98
2. Heated to 143° F. for	4	0.65	6.84	2.104	0.005	3.11	4.78
30 min.	24	0.65	6.82	2.102	0.005	3.34	5.14
3. Heated to 160° F. for	4	0.66	6.83	2.041	0.005	2.64	4.00
5 min.	24	0.66	6.82	2.081	0.005	2.92	4.42

The data in Table 7 show that heat lowers the bound water of pure mik phospholipids. It will be noted again with the temperatures used that the greatest reduction in bound water content occurs between 143° F. and 160° F.

The viscosity of the phospholipid sols decreased on heating to elevated

temperatures. From the results obtained it can be stated that certain high temperatures decrease the bound water content and viscosity of the fat globule "membrane" and pure milk phospholipids.

The Effect of Pasteurization and Homogenization on the Bound Water Content of Twenty-Two Per Cent Cream

A study with cream was undertaken to show the effect of pasteurization and homogenization on the bound water content. A sample of cream was divided into three lots as follows: (1) control of unheated cream; (2) cream heated to 143° F. for 30 minutes; and (3) cream homogenized with a pressure of 2000 pounds at a temperature of 143° F. These data are given in Table 8.

TABLE 8

The effect of pasteurization and homogenization on the bound water content of cream

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Per cent bound water	Grams bound water per gram solids
1. Raw Cream	4	28.53	6.64	7.285	0.15	4.12	0.144
2. Heated to 143° F. for 30 min.	4	28.77	6.66	6.326	0.14	4.01	0.139
3. Homogenized (2000 lbs.)	4	27.83	6.62	28.530	0.135	3.56	0.128

The pasteurization of cream is known to have a detrimental effect upon viscosity and this fact is borne out in Table 8. At the pasteurization temperature used, it is also evident that a small reduction in bound water content occurred.

Homogenization of cream at a temperature of 143° F. and a pressure of 2000 pounds markedly increases the viscosity and decreases the bound water content. The clumping of the fat globules apparently exerts an influence on the protein stability and bound water.

From this study it can be concluded that raw cream contains more bound water and has a higher viscosity than pasteurized cream. Homogenization decreases the bound water, but markedly increases the viscosity.

The Effect of Homogenization on the Bound Water Content of "Ice Cream Mixes"

Some of the effects produced by homogenization in milk and cream are: (1) stabilization of the fat emulsion; (2) destabilization of the protein; (3) increase in surface tension; (4) increase in titrable acidity; or (5) decrease in pH; and (6) increase in viscosity, normally thought to be due to the increase in volume of the disperse phase together with the adsorbed layer and the tendency of the fat to clump.

A "mix" was made containing 14.75 per cent of fat, 12.15 per cent serum solids and distilled water, the sugar and the gelatin being omitted. This "mix" was divided into four portions and treated as follows: (1) homogenized at 0 pounds pressure; (2) homogenized at 1500 pounds pressure; (3) homogenized at 3000 pounds pressure (single valve); and (4) homogenized at 3000 pounds and then at 700 pounds pressure (dual homogenization). The bound water determinations were made in the usual manner and the results are given in Table 9.

TABLE 9

The effect of homogenization on the bound water content of "ice cream mixes"

Mix pressure	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water	Grams bound water per gram solids
1. 0 lbs.	0	26.90	6.43	7.940	0.27	7.4	5.14	0.191
	4	26.90	6.43	9.125	0.27	7.4	5.56	0.207
	24	26.90	6.45	8.829	0.275	7.4	5.29	0.197
2. 1500 lbs.	0	27.01	6.39	12.938	0.275	7.2	2.43	0.090
	4	27.01	6.41	14.123	0.275	7.2	3.22	0.119
	24	27.01	6.42	14.715	0.280	7.2	3.79	0.140
3. 3000 lbs.	0	27.01	6.39	45.293	0.280	7.0	1.96	0.075
	4	27.01	6.41	52.641	0.285	7.0	2.23	0.083
	24	27.01	6.41	53.233	0.285	7.1	3.47	0.128
4. 3000 and 700 lbs.	0	27.01	6.42	10.499	0.275	7.3	5.65	0.209
	4	27.01	6.41	12.799	0.275	7.3	5.93	0.220
	24	27.01	6.41	12.409	0.280	7.4	6.18	0.229

It will be noted from Table 9 that the greater the homogenization pressure (single valve) the greater was the decrease in bound water content and protein stability. It is also apparent that as the viscosity increased due largely to fat clumping (sample 3) there was a decided reduction in bound water content. When the fat clumping and viscosity were decreased by the dual homogenization process (sample 4) a marked increase in bound water content was noted when compared to sample 3.

The fat clumping is undoubtedly largely responsible for the increased viscosity obtained on homogenization, and from the data presented it becomes apparent that the bound water content of these "ice cream mixes" decreases on increased fat clumping and that where dual homogenization is practiced an increase in bound water content and protein stability result.

The Effect of Freezing Milk Plasma on the Bound Water Content

Anderson and Pierce (7) found that after two or three months of storage in a frozen state milk proteins began to precipitate and there seemed to be a slight chemical reaction occurring.

Munkwitz, Berry and Boyer (8) show that freezing causes a partial precipitation of the milk solids; that albumin is precipitated in the greatest

amount, followed in order by lactose, total protein, ash, casein and fat; that with the exception of the fat, the amount of precipitation of the solids increases as the length of time of freezing increases; that freezing causes the fat globules to clump together, and become distorted and irregular in size and shape.

Under the usual conditions of freezing and thawing, the casein of milk, being hydrated, retains its normal degree of dispersion, but when the milk is held in the frozen state over long periods of time, various workers have found that the casein gradually precipitates. It is possible that its hydrophilic properties are changed during storage in a frozen state.

Webb and Hall (9) found that if whole milk is condensed 2½ to 3 times its normal concentration and is then frozen, it may be kept in storage at −13.3° C. (8° F.) or below and reconstituted at any time within about 6 weeks to give a satisfactory fluid milk. Increased milk concentration, higher storage temperatures, or longer periods of storage tended to produce a gelation of the product due apparently to changes in hydration of the casein.

Webb and Hall (10) also found that holding skimmilk in a frozen condition increased the heat stability at 120° C. up to 17 weeks, after which time the heat stability declined. Condensed skimmilk (18% M.S.N.F.) increased in heat stability up to 7 weeks, but subsequently became progressively unstable. They also state that slow freezing of milk or cream caused a gradual precipitation of the caseinate system and an immediate destruction of the fat emulsion.

Doan and Baldwin (11) state that, "Freezing *per se* has no measurable effect on the protein dispersion. Holding in the frozen condition for several weeks or months is required definitely to cause aggregation, denaturation or instability of the proteins."

The effect of long periods of storage in the frozen condition (−15° C.) on the bound water content of skimmilk and condensed skimmilk (2¾ to 1) was studied. Whole milk and cream were also frozen with the intention of determining bound water content but due to destabilization of the butterfat on thawing, accurate solids determinations could not be made and this part of the experiment was therefore discarded.

The samples of skimmilk and condensed skimmilk were frozen and stored at −10 to −15° F. in quart cardboard containers and thawed at room temperature. The skimmilk had been pasteurized at 143° F. for 30 minutes previous to freezing. The results of these experiments are given in Table 10.

The results indicate that holding skimmilk relatively long periods of time in the frozen condition has no great effect upon the alcohol stability of the proteins, but with the condensed skimmilk destabilization occurred. The bound water content of the skimmilk increased slightly and at the 60 day determination it reached its maximum and remained the same up to the 85th day determination. A slight decrease in bound water occurred when determined on the 147th day.

TABLE 10

The effect of freezing milk plasma on the bound water content

Sample	Days frozen	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water
1. Skimmilk	0	9.06	6.55	1.775	0.195	8.5	2.13
	30	9.02	6.45	1.837	0.175	8.5	2.24
	60	9.05	6.43	1.877	0.175	8.5	2.36
	85	9.05	6.43	1.877	0.175	8.5	2.36
	117	9.06	6.48	1.735	0.175	8.3	2.13
	147	9.06	6.49	1.694	0.170	8.2	2.01
2. Condensed Skimmilk	0	25.33	6.15	7.650	0.58	5.4	11.62
	25	25.36	6.05	7.750	0.58	5.5	12.17
	57*	.....	.....	.....	.....	....	.....

* Partial precipitation of the casein on thawing.

The condensed skimmilk increased in bound water when stored in the frozen condition for 25 days. After 57 days a marked precipitation of the proteins occurred. Although bound water determinations could not be made, a decrease in the bound water content probably took place. These results parallel the work of Webb and Hall (10), previously cited, on the heat stability of frozen skimmilk and frozen condensed skimmilk.

The Effect of Added Salts on the Bound Water Content of Concentrated Milk Plasma and of Cream

According to the Sommer and Hart theory of Salt Balance (12), citrates and phosphates usually increase heat stability of milk proteins while calcium and magnesium are usually detrimental in their effect on stability. Since stability is usually associated with the proteins and salt balance, it is reasonable to believe that these stabilizing and destabilizing salts should produce some effect upon the bound water content of dairy products, especially in ice cream mix, evaporated milk and coffee cream.

To the writers' knowledge very little work has been done with the effect of added salts on the hydration of the proteins. The effect of hydration is relatively important in ice cream mixes. If the salt balance is not proper in an ice cream mix there is a tendency for the proteins to precipitate upon homogenization and from the standpoint of coffee cream there is a tendency for the cream to "feather" in the coffee. The use of salts is prevalent in the evaporated milk industry. The proteins of evaporated milk have a tendency to precipitate on sterilization if the salts are not in proper balance.

Sommer (13) shows that proteins can be precipitated by "salting out" and that this process is known to exert a dehydrating effect. He further states that citrates and phosphates have a hydrating effect on casein while calcium decreases its hydration.

Sommer and Young (14) reported that upon adding 0.40 and 0.56 per cent of sodium citrate or 1.26 per cent di-sodium phosphate the ease of whip-

ping ice cream mixes was greatly increased, while 0.1 and 0.2 per cent calcium lactate had a noticeable effect and 0.5 per cent had a marked effect in reducing the ease of whipping of ice cream mixes. Later Sommer (15) found that these results could not always be duplicated.

Hening and Dahlberg (16) added sodium citrate, potassium oxalate (poisonous), and di-sodium phosphate to ice cream before pasteurization, and homogenization and obtained a lower viscosity and a greater whipping ability. Calcium lactate increased viscosity and fat clumping and made the mix more difficult to whip.

Keith, Rink and Weaver (17) found that the citrate ion greatly decreased the viscosity and fat clumping, and decreased the titratable acidity only very slightly. The stability of the proteins was increased particularly toward precipitation by alcohol. The calcium ion gave exactly opposite results from those of the citrate ion.

The effect of sodium citrate and di-sodium phosphate on the bound water content was determined with concentrated milk plasma. The sample was divided into three parts as follows: (1) control; (2) control plus one per cent N/4 sodium citrate; and (3) control plus one per cent N/4 di-sodium phosphate. These samples were pasteurized at 143° F. for 30 minutes, cooled to 40° F. and determinations made, after aging at 40° F., at 4 and 24 hours. The results are given in Table 11.

TABLE 11

The effect of milk stabilizing salts on the bound water content of concentrated milk plasma

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water	Grams bound water per gram solids
1. Control	4	11.87	6.39	2.959	0.24	8.2	4.31	0.363
	24	11.87	6.41	3.020	0.24	8.3	4.64	0.391
2. Sod. Citrate	4	11.81	6.45	3.061	0.225	8.7	4.83	0.409
	24	11.81	6.46	3.122	0.225	8.8	4.95	0.419
3. Di-sodium	4	11.83	6.46	3.020	0.225	8.7	4.95	0.418
Phosphate	24	11.83	6.49	3.102	0.22	8.9	5.42	0.458

The results in Table 11 show that sodium citrate and di-sodium phosphate increase the hydration of the proteins as indicated by a slight increase in bound water content. These stabilizing salts also caused an increase in protein stability. The effects produced on the pH and the acidity do not appear significant although there was a slight reduction in acidity.

The same experiments were repeated using cream instead of milk plasma to note the effects of these stabilizing salts on the bound water content. Table 12 shows the results obtained with these stabilizing salts in cream.

TABLE 12

The effect of milk stabilizing salts on the bound water content of 25 per cent cream

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water	Grams bound water per gram solids
1. Control	4	31.44	6.61	8.641	0.13	7.0	5.90	0.188
	24	31.44	6.63	8.741	0.135	7.1	6.18	0.196
2. Sod. Citrate	4	31.16	6.66	7.857	0.12	7.0	6.01	0.193
	24	31.16	6.71	9.387	0.123	7.15	6.43	0.206
3. Di-sodium Phosphate	4	31.24	6.63	7.714	0.125	7.0	6.04	0.190
	24	31.24	6.68	8.877	0.127	7.2	6.43	0.205

The effect of these stabilizing salts on the bound water content of cream is not marked. The results indicate that these salts do increase the bound water content slightly. It appears, therefore, that the effect produced by sodium citrate and di-sodium phosphate is mainly on the substances in the plasma and not on the substances surrounding the fat globules.

The salts antagonistic in their action upon protein stability are the salts of calcium and magnesium. As a rule they are known to have a destabilizing effect upon the milk proteins. The salts used in this study were calcium phosphate (monobasic) and calcium lactate. These salts were added also on a normality basis to concentrated milk plasma which was then pasteurized at 143° F. for thirty minutes after which it was cooled to 40° F. and determinations made after aging 4 and 24 hours.

TABLE 13

The effect of destabilizing salts on the bound water content of concentrated milk plasma

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water	Grams bound water per gram solids
1. Control	4	14.26	6.32	3.102	0.310	7.7	7.49	0.525
	24	14.26	6.29	3.449	0.310	7.8	7.73	0.542
2. Monocalcium Phosphate	4	14.14	6.26	3.020	0.330	6.8	6.96	0.492
	24	14.14	6.26	3.245	0.330	6.85	7.07	0.500
3. Calcium Lactate	4	14.22	6.29	3.020	0.310	6.8	7.03	0.494
	24	14.22	6.31	3.265	0.313	6.9	7.17	0.504

The hydration of the proteins as measured by the amount of bound water present shows that the so-called destabilizing salts are only slightly detrimental to the bound water content and the stability of the proteins. Both calcium phosphate and calcium lactate decrease the amount of bound water about the same degree. There was also a considerable decrease in protein stability.

The effect of these destabilizing salts on cream was also studied. The

salts were added in the same amounts as previously described and the cream was also pasteurized at 143° F. for thirty minutes.

TABLE 14

The effect of destabilizing salts on the bound water content of 27 per cent cream

Sample	Hours aged at 40° F.	Per cent solids	pH	Viscosity centipoises	Per cent acid	Alcohol no.	Per cent bound water	Grams bound water per gram solids
1. Control	4	33.61	6.51	13.510	0.155	6.0	5.57	0.166
	24	33.61	6.54	16.531	0.160	6.2	5.82	0.173
2. Monocalcium Phosphate	4	33.27	6.44	13.061	0.170	4.7	4.99	0.149
	24	33.27	6.44	14.898	0.170	4.8	5.13	0.154
3. Calcium Lactate	4	33.25	6.58	12.595	0.165	4.5	4.51	0.136
	24	33.25	6.58	15.000	0.170	4.6	4.62	0.138

The effect of destabilizing salts on the bound water content of cream is noted above. Both salts studied appear to decrease the amount of bound water. This decrease in bound water is accompanied by a decrease in viscosity and a decrease in protein stability.

CONCLUSIONS

The bound water content of a prepared casein sol is greatest at a pH of approximately 6.6–6.7 and tends to diminish if the pH is either increased or decreased. Concentrated milk plasma has the greatest amount of water bound at a pH 6.2–6.4 and tends to diminish if the pH is either increased or decreased.

The bound water content per unit of casein tends to diminish with the concentration of the casein sol. Raising the heating temperature lowered the bound water content of the concentrated milk plasma. High pasteurization temperatures decrease the bound water content of the fat globule "membrane" and of pure milk phospholipids.

Pasteurization of cream at 143° F. for 30 minutes decreases only slightly the bound water content while homogenization decreases the bound water content still further. Homogenization decreases the bound water content of "ice cream mixes." The higher the pressure of homogenization the greater was the reduction of bound water content. Destruction of the clumps by dual homogenization increases the stability of the proteins and increases the amount of bound water.

The freezing of skimmilk and condensed skimmilk over long periods of time reduces the protein stability and the bound water content.

The so-called milk stabilizing salts tend to increase slightly the bound water content and protein stability of concentrated milk plasma and cream,

while the destabilizing salts tend to decrease the bound water content and protein stability of concentrated milk plasma and cream.

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JOURNALS

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American Journal of Diseases of Children
American Journal of Physiology
American Journal of Public Health
Archives of Pediatrics

Biochemical Journal
Biochemische Zeitschrift

Canadian Dairy and Ice Cream Journal
Certified Milk
Cornell Veterinarian

Deutsche Molkerei Zeitung

Endocrinology

Food Industries
Food Manufacture
Food Research

Guernsey Breeders Journal
Ice and Refrigeration
Ice Cream Field
Ice Cream Industry
Ice Cream Review
Ice Cream Trade Journal
Industrial and Engineering Chemistry

Jersey Bulletin
Journal of Agricultural Research
Journal of Agricultural Science
Journal of American Chemical Society
Journal of American Veterinary Medicine Association
Journal of Bacteriology
Journal of Biological Chemistry
Journal of Dairy Research
Journal of Dairy Science
Journal of Experimental Medicine
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Journal of Milk Technology
Journal of Nutrition
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Kolloid-Zeitschrift

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Milchwirtschaftliche Zeitung
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Milk Industry
Milk Plant Monthly
Molkerei Zeitung

National Butter and Cheese Journal

Pacific Dairy Review
Proceedings of Society of Animal Production
Proceedings of Society of Experimental Biology and Medicine

Tierernährung
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Zeitschrift für Infektionskrankheiten Parasitäre Krankheiten und Hygiene der Haustiere
Zeitschrift für Physikalische Chemie, Abt. A and B
Zeitschrift für Untersuchtung der Lebensmittel
Zeitschrift für Züchtung. Reihe B. Tierzuchtung und Zuchtungbiologie
Zentralblatt für Bacteriologie
Züchtungskunde

SPECIAL PUBLICATIONS

Federal Dairying and Bacteriological Establishment, Liebefeld, Berne, Switzerland

International Association of Ice Cream Manufacturers
International Association of Milk Dealers

National Institute for Research in Dairying, Reading, England
New York Association of Dairy and Milk Inspectors

Prussian Dairy Research Institute, Kiel, Germany

State Agricultural Colleges and Experiment Stations

The Royal Technical College, Copenhagen, Denmark

United States Department of Agriculture

ABSTRACTS OF LITERATURE

CHEMISTRY

267. A New Method for the Determination of Butterfat in Dairy Products. JOHN GOLDING. J. Dairy Research *8*, p. 275, 1937.

A simple gravimetric method suitable for the determination of fat in cream, ice cream mix, milk, and possibly other dairy products is described. The fat is separated by churning following the addition of a variable quantity, dependent on the product being tested, of a reagent consisting of 75 ml. of C. P. ammonium hydroxide, 35 ml. of n-butyl alcohol, and 15 ml. of 95 per cent ethyl alcohol. The butter is washed with water and then removed to a metal dish for frying. The percentage of fat is calculated from the weight of dried fat and the weight of the original sample. Results were found to agree with the Roese-Gottlieb analysis within $\pm$ 0.05 per cwt.

S.T.C.

CONCENTRATED AND DRY MILK; BY–PRODUCTS

Abstracts of interest are numbers 267 and 271.

DISEASES

268. Bovine Mastitis. III. A Comparison of the Bacteriological Reactions of Normal and Mastitis Milk from Young Cows. RALPH B. LITTLE, The Rockefeller Inst. for Med. Research, Princeton, N. J. Cornell Veterinarian *28*, 1, p. 23, Jan., 1938.

The milk from eight first calf heifers was examined before and after their udders were infected with a double zone hemolytic streptococcus. The methods used were daily examinations of chlorine, leucocyte count, bacteriological examination of the milk, and pH values. After the onset of subclinical mastitis in these eight animals, 2163 daily examinations of the fore milk showed that the bacteriological plating of the milk in blood agar was more efficient in the detection of infection than any other method employed for typical hemolytic streptococci were observed in the milk of every examination. The next more reliable test was the direct leucocyte count; in 94 per cent of the examinations the count per cc. of milk was between 300,000 and 10,000,000 cells or over.

L.A.M.

269. Detection of Mastitis by the Bromthymol Blue Test, Leucocyte Count, and the Microscopic Examination of Incubated Milk. A. C. FAY, H. W. CAVE AND F. W. ATKESON, Kan. Agr. Exp. Sta., Manhattan, Kan. Cornell Veterinarian *28*, 1, p. 40, Jan., 1938.

On a basis of routine examination of individual quarter samples of milk from 114 cows in the college herd the animals were segregated into three

principal classes: Class A, 70 animals regarded as free from mastitis; Class B, 17 head regarded as suspicious for mastitis because of high leucocyte count (500,000 or more per cc.) in the milk from one or more quarters; Class C, 27 head, regarded as positive for mastitis because of presence of long chained streptococci in samples incubated 16 hours and usually though not necessarily a high leucocyte count. The results of the studies showed that although the Bromthymol Blue test rarely gives a false reaction with a known negative cow, it fails to detect a sufficiently high percentage of the opposite cows to recommend it as a sole means of identification of mastitis for segregation purposes. The fact that high leucocyte counts above the arbitrary standard of 500,000 per cc. were found in 36.7 per cent of the samples containing long chained streptococci suggests that the standard is too high for proper interpretation. Leucocyte counts above 100,000 per cc. and the appearance of streptococci in incubated samples of milk in chains of only medium length frequently gave fore warning of impending mastitis. It was found inadvisable to move a cow from an infected group to the non-infected group because of a few negative tests. L.A.M.

270. Infectious Bovine Mastitis. 5. Bovine Mastitis and Milk Yield. G. C. WHITE, E. O. ANDERSON, R. E. JOHNSON, W. N. PLASTRIDGE, F. J. WEIRETHER, Storrs Agr. Exp. Sta., Storrs, Conn. Bul. 220, August, 1937.

To determine the effect of mastitis on milk yield data are studied on 35 cows in the Connecticut State College dairy herd and on 22 cows in a farmer-owned herd. Since in both herds animals with acute cases were discarded, the study deals chiefly with incipient and mild forms of the disease.

Four tests are used to determine the status of animals during the various lactations, namely: identification of organisms, bromthymol blue test, leucocyte count, and sediment test.

Lactations covering 240 days corrected to full age and to three time milking for the college herd and two time milking for the farmer's herd, are used to compare production before and after reaction. In the college herd a loss of 679 pounds of milk or 6.5% and in the farmer's herd, of 559 pounds of milk or 5.7% resulted from infection. The amount of loss increased with the number of quarters affected, reaching 1134 pounds or 12.0% and 1111 pounds or 11.2% respectively in the college and the farmer's herd, when all four quarters reacted positively.

Individual lactation period curves covering four or more lactations are shown for five animals and compared with the normal expected production curve.

About three-fourths of all animals show a loss in yield following reaction. Some individuals may pass through several lactations before yield is drastically reduced, or the milk becomes abnormal in appearance; some show a

loss for one or two lactations, and then recover in yield; others suffer a drastic loss in yield and may continually produce abnormal appearing milk. The evidence shows no shortening of the lactation period.

No evidence was found to indicate that mastitis decreases the percentage of fat in the milk, the average tests being 4.16 before and 4.15 following reaction. A.I.M.

FEEDS AND FEEDING

271. The Relative Values of Raw and Pasteurized Milk in the Feeding of Calves. J. Wilkie, S. J. Edwards, A. B. Fowler and N. C. Wright. J. Dairy Research *8*, p. 311, 1937.

Bull calves from tuberculin-tested Ayrshire herds were fed on raw or commercially pasteurized milk up to 12 weeks of age, in amounts in relation to their body weights. This diet was supplemented from the eighth week by hay at the rate of 2/3 pound per head per day. The milk used was mixed milk from untested herds, the raw and pasteurized milk being derived from the same bulk samples.

No significant differences were noted in gain in live weight or in skeletal growth. Marks awarded by experimental stock judges showed consistent differences in favor of the pasteurized milk-fed group.

Inoculations of grouped daily aliquots of raw milk twice weekly into duplicate guinea-pigs resulted in finding viable tubercle bacilli in 70 per cent of the samples and *Brucella abortus* in 38 per cent of them. The pasteurized milk samples were uniformly negative to both tests. The differences in tuberculous infection of the two types of milk were reflected in the results of tuberculin tests and post-mortem examinations on the calves. Twenty-four out of thirty-six calves fed on raw milk reacted to the test. One calf in the pasteurized milk-fed group reacted to the test, but post-mortem examination and inoculation of glandular material into guinea-pigs failed to confirm the presence of any tuberculosis. S.T.C.

272. Further Studies on the Influence of Green Fodder, Silage and Hay on the Metabolism of Ruminants. Fritz Schnepf, Tierzuchtinstitut der Albertus-Universität Königsberg. Biedermann's Zentralblatt, Abteilung B: Tierernahrung. *9*, 3, p. 191, 1937.

These studies were planned to determine (1) the relationship between Ca metabolism and pH of the urine on one hand, and CO_2 tension of the blood plasma and pH of the blood on the other hand, and (2) the effect on the animal body of green feeds preserved in different ways.

Wethers were fed a mixture of second cutting red clover and timothy as green plants, as hay, and as silage. The hay was dried on racks. To the silage was added as a preservative for each 100 kg. of green crop (1) ten acid equivalents of HCl, (2) ten acid equivalents of H_2SO_4, (3) one per cent of sugar.

Results indicate that:

(1) CO_2 tension is as good a criterion of the potential alkalinity or acidity of a feed as is the Ca metabolism; the pH of the blood is too constant to be of any value for indicating small differences; the pH of the urine is influenced markedly by the nature of the ration, but individual differences are often greater than species differences.

(2) Hay has a stronger basic effect than green plants.

(3) Silage preserved with sugar has about the same effect on acid-alkali balance in the body as hay or green plants.

(4) Silage preserved with HCl or H_2SO_4 exerts a distinctly negative effect on Ca metabolism and the blood picture.

(5) If about one-fourth of the dry matter is in the form of hay, sometimes restoration of balance is noted, but it is not enough to compensate the total effect of the mineral acids. J.G.A.

273. Utilization Experiments on Ruminants with Artificially Dried, Chopped Protein-Rich Green Fodders. G. Frölich und F. Haring, Institut für Tierzucht und Molkereiweisen der Martin-Luther-Universität Halle a.S. Biedermann's Zentralblatt, Abteilung B. Tierernahrung. *9*, 3, p. 204, 1937.

Digestibility by wethers of the nutrients of three dried products prepared from green alfalfa and from vetch mixture (so-called Landsberg mixture) by the Rema Rosin drying method, is reported.

Digestibility of artificially dried green alfalfa was improved by grinding as compared with chopping. The chopped vetch mixture was somewhat more digestible than the dried alfalfa. The following values are reported:

Digestible crude protein—in alfalfa meal, 10.83%; in chopped alfalfa, 10.35%; in chopped vetch, 10.17%; starch values—29.33, 26.90, and 38.52% respectively. J.G.A.

274. Significance of Cod Liver Oil in Calf Feeding. Lauri Paloheimo, Institut für Haustierlehre der Universität Helsinki. Biedermann's Zentralblatt, Abteilung B. Tierernährung. *9*, 3, p. 234, 1937.

In an earlier paper (this journal, *9*, 52) an account was given of work in which Ayrshire calves were reared with small quantities of whole milk, their requirements for fat-soluble vitamins being supplied by addition of cod liver oil.

The work has been continued, cod liver oil being omitted, other conditions being the same as already noted. Maximum gains during the first few months were not striven for but the calves were well cared for, and were in good health throughout. Indigestion was specially guarded against.

Under these conditions, even when the total quantity of whole milk was limited to 10–15 kg., calves seemed to thrive nearly as well as when 5–10

grams of cod liver oil were fed daily. The dams, with two exceptions, had received feeds rich in vitamins during the latter part of the gestation period. But the two exceptions indicate (but two calves are too few to prove) that this preparatory feeding of the dams is not absolutely necessary for thrifty calves, even if these are reared with very little whole milk and without cod liver oil. J.G.A.

275. Studies on the Influence of Air Tight Covers on the Preservation and Value of Silage. KURT DIETRICH, Tierzucht-Institut der Albertus Universität Königsberg. Biedermann's Zentralblatt, Abteilung B: Tierernährung. *9*, 3, p. 255, 1937.

Glass cylinders were filled with chopped clover or marrow stem cabbage. To one lot no preservative was added; others had sugar or sulfuric acid added. The cylinders were closed with rubber stoppers each provided with a fermentation tube. A parallel series were sealed with a layer of loam. Larger scale trials were conducted in Aurich fermentation vessels of 1 cubic meter capacity, or in Tschechnitz fermentation chambers.

Conclusions reached were:

1. Complete exclusion of air hinders fermentation, as indicated by high pH values, very little lactic acid formation, a large amount of combined acetic acid, and formation of a small amount of butyric acid.

2. Wilting before ensiling still further slows up the process with clover; with m.s. cabbage more compact storage and corresponding improvement in the silage was noted.

3. Sugar favorably affects the ensiling of fresh plants as well as of wilted ones.

4. Chopping effected an improvement in all cases.

5. Addition of CO_2 has a favorable effect, but this is small in comparison with the effect of sealing the chamber with a layer of loam.

6. Addition of mineral acids checks the formation of CO_2 almost completely.

7. Comparison of the silage from air tight vessels with that from similar vessels covered with a layer of loam indicates that in all cases the upper layers were superior when the latter method was used.

8. There were no considerable differences in protein degradation in the several lots.

9. Digestion trials (wethers) do not indicate any superiority of one method over another. J.G.A.

HERD MANAGEMENT

276. Biometrical Study of the Production Improvement in a British Friesian Herd. HANS LÖRTSCHER, Zeitschr. Züchtung. Reihe B. Tierzücht. u. Züchtungsbiol. *39*, 3, p. 257, 23 fig., 1937.

The milk production records from 1906 to 1932 in a large Friesian herd in England were studied to measure the non-hereditary factors influencing milk yield and to find ways to correct for those factors when studying inheritance or making selections. About 4000 lactations from some 1200 cows were included. A multiple regression equation and nomogram were developed for standardizing the test-year records for age of the cow when the test year began, for length of the dry period, for length of the service period, and for the inter-relations between these factors. The length of the preceding dry period was of slight importance unless it was shorter than three weeks. The month of calving, the sex of the calf, and whether it was a twin or a single birth had little effect. Expressing each individual yield relative to the herd average for the same year (the "Stalldurchschnitt" method of Peters, v. Patow, Krüger, and others) was studied as a means of correcting for intangible environmental factors such as weather, changes in feeding policy, etc. Complete reliance on these relative figures would imply that all year-to-year differences in the herd average are of environmental origin. On the contrary the author finds that in his material many of the differences in the yearly averages resulted from changes in the average genetic composition of the herd. This conclusion is based on the steadiness of the trends and on comparing the year-to-year changes of the herd average when only those cows which had records in both years were included, with the contemporary changes in the herd average when it included the records of all cows. In this procedure the author offers a way to use the herd average to correct for unrecorded changes in herd environment, yet without assuming that the average genetic composition of the herd is unchanging. That the method will also furnish a bridge for comparing the records of two cows in different herds unless those two herds happen to have interchanged a number of cows, is not apparent.

The author concludes that 50 to 70 per cent of the variance in the milk records with which he worked (corrected for age, dry period and service period) is genetic, but that construes as genetic nearly all of the differences in yearly averages. Those differences account for nearly 24 per cent of the total variance in individual records. Regression of individual productions on changing conditions is thought not to be entirely linear and not to follow the same course for all cows. This makes Mendelian analysis difficult, if possible at all.

This particular herd was outbred in its early history, the breeding stock coming from various other British Friesian herds; then it was outbred with stock imported from Holland; finally there was a period of breeding entirely within the herd, with linebreeding directed toward the imported animals but with efforts to avoid inbreeding while staying within the herd. The inbreeding did not become high enough to produce noticeable effects. Coefficients

of inbreeding and *inter se* relationship are presented to make the description of the breeding policy of the herd more complete and quantitative.

J.L.L.

277. Measuring Milk Producing Ability. L. KRÜGER. Breslau. Züchtungskunde *13*, 1, p. 2, Jan., 1938.

The author emphasizes the distinction between the milk producing *ability* of a cow and the *record* of the milk she actually produced. His method of correcting for differences in environmental circumstances is presented and its use is illustrated with a few examples. Yields are corrected to a standard interval of 360 days between calvings. The last 50 days of this interval are a dry period. Correction is also made for deviations in the length of the preceding calving interval and dry period and for age. Corrections for unrecorded environmental conditions applying to that herd are made by multiplying the record of this cow by the ratio of the standard production in that region for that system of dairying to the average production of the cows which freshened in the same herd during the six months centering around the time that this cow freshened. The inherent assumption, that the average producing ability of the group of cows freshening in those six months in that herd is equal to the average producing ability of all cows of that region, doubtless has exceptions and needs some qualifications. If those can be had, this process of correction offers an automatic way of correcting for general conditions of management and feeding, without recording what those were nor estimating the effect of each. There is, of course, need for judgment in deciding what groups of herds should be combined to provide the standard for that region and for that type of agriculture. There is also opportunity to exercise judgment in discarding records which were "abnormal" for reason of accident or distinct and unmistakable illness. J.L.L.

ICE CREAM

278. A Summary of Six Years of Research on Tallowy Flavor in Strawberry Ice Cream. C. A. IVERSON, Iowa State College, Ames, Iowa. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 7, Oct., 1937.

The author concludes from this study that oxidases from the fruit are not responsible for causing oxidized flavors in strawberry ice cream.

The higher the iron content of strawberry ice cream, the less oxidized flavor developed. This was thought due to the existence of ferrous iron in combination with a milk constituent, serving in this combination as an antioxidative catalyst.

Reichart-Meissl numbers and acetyl values determined on fat extracted from ice cream showed no relation to the rate of development of oxidized

flavors. The trend in the iodine number changes indicated fat oxidation during development of oxidized flavors.

Oxidized flavors developed more frequently when whole or skim condensed milk were used in mixes as compared with dry skimmilk or condensed skim or whole milk made in stainless steel pans.

The author concludes, further, from experimental evidence that the addition of fruits to ice cream actually retards the development of oxidized flavors in ice cream.

The copper content of mixes in which no oxidized flavors developed was found to be less than 1.18 p.p.m., whereas the lower limit of the copper content of ice creams which became oxidized was 1.80 p.p.m. When mixes contained copper contents between these limits and developed oxidized flavors, it was assumed that factors other than the copper content contributed to the development of the off-flavor. It is also stated that the gross quantity of copper present is not the only factor of importance, but the state in which the copper exists in the ice cream is also of significance. M.J.M.

279. Quality Through Freshness. A Discussion led by H. F. JUDKINS, National Dairy Prod. Corp., New York, N. Y. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 104, Oct., 1937.

(a) A. C. BITTER, Wm. Neilson, Ltd., Toronto, Can.

Packages are marked in code and each driver is equipped with a key. It is his responsibility to see that the oldest stock is sold first in the stops along his route. By limiting the number of articles and eliminating slow selling products in winter months it was possible to carry only five standard bricks and a weekly special. By so doing, fresher ice cream has been sold and sales have increased considerably.

(b) J. A. CLUTTER, Dairyland, San Antonio, Texas.

In each retail store some ice cream was set aside, held for three weeks, and then scored against the fresh product. On the average, the ice cream had deteriorated about two points in flavor score over the storage period of three weeks. If fresh high quality ingredients are used to make a desirable product, it should be marketed promptly before the ice cream deteriorates in quality.

(c) GEORGE A. KURK, Beatrice Creamery Co., Lincoln, Neb.

The necessity of selling fresh ice cream has been shown repeatedly. In order to achieve this, careful supervision of the hardening room is essential, a survey of the dealers' sales possibilities is desirable, and lastly many companies should eliminate small accounts where the daily sales are low and the ice cream deteriorates before being sold.

(d) R. J. QUIRIE, United Farmers Cooperative Creamery Assn., Charlestown, Mass.

The consumer often does not get the same high quality ice cream which is placed in the cabinet of the distributor. The answer lies in what might be called sales or service contact work which can be done by the route salesman, the territory salesman or one especially designated for the work. The distributor's stock should be checked, the mechanical and sanitary character of the cabinet should be watched and the cabinet should be operated at a sufficiently low temperature for packaged goods.

(e) J. FRANK WARD, Midwestern Dairy Products, Salt Lake City, Utah.

Much of the trouble with off-flavors is due to the use of inferior products as well as the failure to market ice cream promptly. Both factors must be watched.

The operation of retail ice cream stores by ice cream manufacturers has done much to teach the manufacturer the kind of ice cream the public prefers.

SUMMARY BY MR. JUDKINS

Data were presented to show the variance in rate of turnover for one dealer as compared to another. Bulk sales varied from 2 to 39 gallons per gallon of inventory for the period of October 1 to April 1, 1934. The variation in sales of brick ice cream was given for the same period, as well as the distribution of products stocked in the cabinets. From this information the following recommendations were made:

1. The sales department should study the relation of sales volume to flavors of bulk and package goods carried.
2. Brick package units such as one-half gallon units, should be kept in the cabinet properly.
3. Drivers should be instructed regarding the proper use of these units and should educate the dealers how to use them. M.J.M.

280. The Statistical and Accounting Bureau. O'NEAL M. JOHNSON, Intern. Assoc. Ice Cream Mfgrs., Washington, D. C. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *3*, p. 7, Oct., 1937.

During the year the Statistical and Accounting Bureau released three bulletins on taxation. The Association Accounting system was installed in a considerable number of plants. Other activities of the bureau included a report of the survey of the retail store, the advertising analysis and the analysis of ice cream sales, and the ice cream sales index for 1936. Preliminary reports of sales in 1937 have also been released. M.J.M.

281. The Accountant Looks Toward 1938. J. S. BLOOM, Abbotts Dairies, Inc., Philadelphia, Pa. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *3*, p. 10, Oct., 1937.

It is the accountant's job to ascertain the trend in cost of raw products, manufacturing and labor costs, taxes, sales, new demands in sanitation and the trend in other costs as well. It is necessary to chart the course of busi-

ness if comptrollers and accountants are to serve the best purposes of the industry. M.J.M.

282. Controlling Advertising Costs. K. R. Leach, Dairymen's League, Inc., Syracuse, N. Y. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *3*, p. 16, Oct., 1937.

The proper control of advertising costs depends upon:

1. The setup of a budget and advertising plant at the beginning of the year.
2. The adherence to this budget and plan during the year.
3. The constant checking of the effectiveness of advertising both from an expense and a result point of view.
4. Proper checking of expenditures as to accuracy of charges made against the advertising account.
5. A preparation of analysis for study by management to enable management to plan its future advertising programs.

These five things should form a basis for proper control of advertising and should increase the effectiveness of the advertising. M.J.M.

283. Controlling Selling Costs. H. W. Brigham, Teall's Ice Cream Co., Rochester, N. Y. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *3*, p. 22, Oct., 1937.

The control of selling costs is more or less controlled by the policy set by the management. It is possible in the larger company with a large sales force to consolidate territories and thus save expense. However, the company with three or four men traveling finds it almost impossible to vary the budget for sales expenses without altering the effectiveness of the work. M.J.M.

284. Controlling Trucking Costs. H. W. Schyelke, Southwest Ice and Dairy Products, Oklahoma City, Okla. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *3*, p. 30, Oct., 1937.

Selling and delivery expenses in many businesses have been steadily mounting. The qualifications of the driver salesman affect the trucking costs on his route. The type of delivery service and the kind of delivery equipment enter materially into determining trucking costs. Telephone expenses and special delivery service often are allowed to increase costs unnecessarily.

The truck salesman should keep a detailed expense account and the adherence to this record keeping often helps to show him the necessity to keep costs lower when possible. M.J.M.

285. Controlling Cabinet Costs. J. E. Shipley, Abbotts Dairies, Inc., Philadelphia, Pa. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *3*, p. 34, Oct., 1937.

By keeping simple monthly accounts of cabinet service work done, repair shop overhead, truck costs, and expenditures for materials, it is easy to determine cabinet service costs. With this information it is possible to decide whether it would be cheaper to hire an outside cabinet service contractor. The data should also show whether it is cheaper to rebuild or to buy new equipment; or if repairs increase, the real cause of the increase can be determined. The need for having adequate records so that the causes for changes in costs can be determined is imperative. M.J.M.

286. What Management May Expect from the Association Accounting System. C. A. ARMITAGE, United Farmers' Coop. Creamery Assn., Charlestown, Mass. Proc. 37th Ann. Conv. Int. Assn. of Ice Cream Mfgrs. *3*, p. 42, Oct., 1937.

There are many services one should expect from the association's accounting system. It should show when new equipment should be bought, what the operating costs should be, and what savings should be made. The system shows fluctuations in expenses and furnishes a basis for the investigation of these expenses. Such a study should be made at regular intervals. The system provides a basis for determining the costs of products and production and in this way makes it possible to ascertain the necessary selling price for each item. By showing which items are most profitable the accounting system shows which products to feature.

Intelligent merchandising is impossible without knowing unit costs for each product made. The association accounting system furnishes this information. M.J.M.

287. Proved System Short Cuts. O'NEAL M. JOHNSON, Intern. Assoc. Ice Cream Mfgrs., Washington, D. C. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *3*, p. 47, Oct., 1937.

A number of changes and additions to the Association Accounting System which have increased the effectiveness of the system were explained. This was followed by a discussion of problems and suggestions pertaining to accounting. M.J.M.

288. Cutting Office Costs. J. S. BLOOM, Abbotts Dairies, Inc., Philadelphia, Pa. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *3*, p. 65, Oct., 1937.

Since the main expense in office costs is labor, the main problem in controlling the costs is to study labor costs. There are different grades of labor to be done in an office and a person trained and fitted for each grade should be employed. A program of work should be planned for each person in the office.

Other important considerations are to have the necessary mechanical appliances for most efficient work from the employees of the office, and the

office should be so organized that the flow of work from person to person is accomplished efficiently. M.J.M.

289. Late Developments in Hardening Room Installations: Bay Type of Vertical Coils. RIDGWAY KENNEDY, JR., Abbotts Dairies, Inc., Philadelphia, Pa. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 20, Oct., 1937.

The ammonia coils used in the hardening room were installed in a vertical position in bays or bins and the ice cream was stacked in the bays. A coil unit was developed which could be fabricated and welded at the factory. Each bay was of the same size and 175, 2½ gallon cans were stacked in each bay. The article contains photographs and figures and complete instructions for the construction of this type of hardening room.

There are several advantages of the bay type of installation. Defrosting proved unnecessary because the frost was shaken or rubbed off the coils by piling the cans of ice cream against the coils. The amount of pipe in the room was reduced from forty thousand feet of two-inch pipe to twenty-five thousand feet of one and a quarter inch pipe. The refrigeration is utilized more efficiently. Less difficulty from ammonia leaks has been experienced. M.J.M.

290. Problems in Meeting Bacterial Standards for Ice Cream. H. MACY, Univ. of Minnesota, St. Paul, Minn. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 36, Oct., 1937.

Sanitary codes adopted by most states have bacterial standards which range between 100,000 and 500,000 per milliliter. However, certain cities have adopted maximum bacterial standards as low as 25,000 per milliliter. Considerable vigilance is necessary to meet such exacting requirements.

The problems of meeting bacterial standards involves three issues, namely, the procurement of high quality ingredients, the effective pasteurization of the mix, and the maintenance of proper sanitary conditions within the plant at all times. When these issues are satisfactorily met, the most exacting standards may be regularly observed.

An efficient laboratory service is essential if bacterial counts are to be controlled successfully. Constant checking of materials, plant processes and sanitary measures is essential. If the cost of such service seems prohibitive for the smaller companies, thought should be given to the formation of cooperative laboratory service. M.J.M.

291. Proper Butterfat Differentials between Fruit and Vanilla Ice Cream. CARL KOERVER, The Borden Co., Brooklyn, N. Y., AND HAROLD PRATT, Philadelphia Dairy Prod. Co., Philadelphia, Pa. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 43, Oct., 1937.

Fat differentials between fruit and vanilla ice creams for the various states are given. In the majority of instances a differential of 2 per cent in fat content is allowable. In order to comply with the standards and, at the same time, use a high percentage of fruit many companies are forced to prepare a special mix for fruit ice cream.

As high as 32 per cent fruit has been used in making certain fruit ice creams. Starting with a 12 per cent mix, the fat content of the resulting ice cream would be 8.16 per cent. The reduction in fat is nearly double the differential allowed by law in many states.

Many states also have standards for total milk solids. Two states permit no reduction of the milk solids for fruit or nut ice cream; the balance permit a reduction of 2 to 5 per cent. Using 30 per cent fruit, a reduction of 6 per cent milk solids should be permitted.

The authors favor a differential between vanilla and fruit ice creams in state standards of 4 per cent fat and 6 per cent total milk solids. They urge that individual manufacturers, as well as associations see that their interests are properly protected by advocating sound regulations. M.J.M.

292. A Simplified Solids Tester for Ice Cream Mix. KENNETH M. RENNER, Texas Tech. College, Lubbock, Texas. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 52, Oct., 1937.

A method is described and the necessary equipment is listed for a simplified solids tester for ice cream mix. The cost of the equipment, exclusive of a Torsion Balance Moisture Scale, is approximately 12 dollars. Results given indicate that the method checks within 0.25 per cent of the Mojonnier test in every instance where comparisons were made. When used with plain condensed skim milk, results similar to those with ice cream mix were secured, but with evaporated milk the results averaged 0.15 per cent higher than the Mojonnier method. M.J.M.

293. Consumer Preference: (a) A Study of Ice Cream Types. P. H. TRACY, Univ. of Illinois, Urbana, Ill. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 60, Oct., 1937.

The following conclusions were drawn from a large number of consumer-tasting tests of ice cream:

1. Body and texture are very important factors in influencing consumer preference for ice cream. Consumers preferred an ice cream having a smooth body and texture.
2. The flavor of a medium fat ice cream was preferred to that of a high fat content.
3. The high serum solids ice cream was preferred to the medium serum solids ice cream.
4. Approximately 25 per cent of the testers preferred a homemade type of ice cream to the regular commercial type of ice cream.

5. From the standpoint of this test a stabilizer is a desirable constituent of ice cream.

6. Ice cream containing a medium yellow color was preferred to uncolored ice cream.

7. The preference for vanillas varied greatly.

8. The medium and high sugar content ice cream was preferred to a low sugar content ice cream.

9. A greater percentage of women than men showed a preference for ice cream with a pronounced flavor and a heavy body. M.J.M.

294. Consumer Preference: (b) Effect of Serving Temperature. W. H. E. REID, Univ. of Missouri, Columbia, Mo. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 72, Oct., 1937.

The results of this investigation indicate that the serving temperature is a factor of considerable importance in determining consumer acceptance of ice cream and sherbets. Dipping properties and the stability of the products studied are also affected by the serving temperature.

The most desirable temperature for serving most ice cream and sherbets was 10° F. However products with mild flavors or a low sugar content were preferred at a higher temperature than 10° F. and those with strong flavors or high sugar content were more acceptable at temperatures below 10° F. The flavor became more pronounced in all ice creams and sherbets as the temperature was increased from 6° F. to 18° F.

The body of both ice cream and sherbets was termed too resistant at 6° F. Sherbets were criticized at 14° F. or higher as being soggy and lacking in resistance. The same criticism was made of ice creams served at 18° F.

M.J.M.

295. Sanitary Factors at the Fountain other than Ice Cream. F. W. FABIAN, Michigan State College, East Lansing, Mich. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 90, Oct., 1937.

A survey was made of twenty-one drug stores and twenty-nine restaurants and dairy bars in an attempt to study from a bacteriological viewpoint each item entering into the serving of ice cream.

The work indicates that there should be a bacterial standard for dipper water and that a satisfactory standard would be the same as the maximum permissible bacterial count allowed for ice cream.

Of the flavors and syrups tested, chocolate syrup usually was the heaviest contaminated and contributed the most bacteria to a dish of ice cream. Strawberry, cherry, pineapple, peach, butterscotch, lemon and orange syrups were sources of contamination, they are listed in the order of the average bacterial content. A bacterial standard of 5,000 bacteria per cc. is proposed for the flavoring and fruit syrups used with ice cream.

Unless proper precautions are observed, ice cream can be grossly contaminated during serving. Dipper water contamination usually is greater

than that from chocolate and fruit syrups. Spoon and dish contamination are negligible if they are washed in soapy wash water at 110 to 120° F. containing less than 50,000 bacteria per cc., then rinsed in water at 170° F. for at least a minute or for a minute or longer in clean water containing 50 to 200 p.p.m. of available chlorine.

Forty-six per cent of the ice cream sold in Lansing and East Lansing, Michigan, was found to exceed the legal bacterial standards of 150,000 bacteria per cc. M.J.M.

296. A Cooperative Quality Improvement Program. E. H. Parfitt, Purdue Univ., Lafayette, Ind. Proc. 37th Ann. Conv. Intern. Assoc. Ice Cream Mfgrs. *2*, p. 100, Oct., 1937.

The quality improvement program sponsored by Purdue University for Indiana ice cream manufacturers was presented. The results of the analysis and scoring of ice cream samples was given in tabular form. The success of the project is indicated by the increased number of commercial samples submitted each year. M.J.M.

297. Best Selling Flavors. Ice Cream Trade J. *34*, 1, p. 9, Jan., 1938.

Information collected by Esmund, Gundlach and Co., Cincinnati, Ohio, from large and small manufacturers located in all parts of the United States and Canada, indicated that the 12 most popular flavors of ice cream were vanilla, strawberry, chocolate, cherry, maple nut, orange pineapple, butter pecan, black walnut, banana, peach, lemon, and pineapple. Next to the above came chocolate variations, fruit salad, almond toffee, butterscotch, peppermint stick, tutti fruitti, caramel, and pecan krunch. Sherbets were rated as follows: orange, pineapple, raspberry, lemon, and lime. W.H.M.

298. The Year's Research Record. J. C. Hening, New York Agr. Exp. Sta., Geneva, N. Y. Ice Cream Trade J. *34*, 1, p. 24, Jan., 1938.

Subjects discussed briefly are: homogenization by sonic vibration, a new vacuum method of freezing fresh milk and cream for use in ice cream, problems encountered in making ice cream of a high butterfat content, the use of cerelose in ice cream, comparison of different serum solids concentrates for ice cream, sodium alginate as a stabilizer for ices, *E. coli* as an index of pasteurization efficiency, sanitary procedure for handling ingredients added to the mix after pasteurization. W.H.M.

299. How to Figure Mix Costs. E. L. Reichart, The Univ. of Nebraska, Lincoln, Nebr. Ice Cream Trade J. *34*, 2, p. 22, Feb., 1938.

The cost of ice cream mix varies greatly depending upon the cost and availability of the various ingredients used and upon the composition of the mix. Generally a plant with an output of less than 20,000 gallons annually is better off to buy mix. In territories where fresh products are available,

mixes can be best and cheapest made using milk, cream and condensed skim as sources of fat and serum solids. In localities removed from actual production areas, mixes are cheapest when made from butter, skimmilk powder or other concentrates. W.H.M.

300. Sales Increase 300 Per Cent. Lucious S. Flint. Ice Cream Trade J. *34*, 2, p. 12, Feb., 1938.

Details are given for a sales campaign used by Edy's Grand Ice Cream Company, Oakland, California, which has materially increased the amount of ice cream sold to their dealers. The plan is built around a monthly feature special and the use of point-of-sale display material. Carefully worked out receipts are furnished the dealers and a great deal of personal contact work is done to assure the success of the campaign. W.H.M.

Other abstracts of interest are numbers 267, 271, and 303.

MILK

301. Bitterness and Thinning in Canned Cream. A. A. Nichols, G. R. Howat and C. J. Jackson. J. Dairy Research *8*, p. 346, 1937.

Three organisms, all strains of *Bacillus subtilus,* were isolated from defective commercially-canned cream which, on inoculation into normal cream, caused bitterness and thinning. Some thirty-six strains of *B. subtilus* were isolated from canned dairy products but only a few were capable of producing the defects when inoculated into normal cream. The spores of these organisms were found capable of withstanding temperatures up to 120° C. for as long as 40 minutes. The defects developed more rapidly by incubation at 37° C. than at lower temperatures. Determinations of the non-protein nitrogen and of the peptone and sub-peptone fractions indicated that the development of both bitterness and thinning were related to the breakdown of the protein. Owing to the high thermal death point of the organisms responsible for the defects, it was expected that control under commercial conditions must depend more on improving the quality of the incoming milk supply than on altering the conditions of sterilization of the canned product. S.T.C.

302. Soft Curd Milk of the Mineral Modified Type. Anonymous. Milk Dealer *27*, 6, p. 43, March, 1938.

A brief description of soft-curd milk of the Mineral modified type and its sale in Chicago. C.J.B.

303. The Problem of Recontamination of Pasteurized Milk and Its Products. L. C. Bulmer, Bureau of Food and Dairy Inspection, Jefferson Co. Bd. of Health, Birmingham, Ala. Milk Dealer *27*, 6, p. 76, March, 1938.

Based on a study of conditions which prevail in all the 51 major cities of America, with population over 200,000, this paper deals not only with the vexed problem of the promiscuous handling of pasteurized milk and ice-cream mix, but also the relationship between boards of health and industry at the present time.

The author also points out that Birmingham is the only city in America that has regulations which adequately control this problem. C.J.B.

304. Sweet Acidophilus Milk. Anonymous. Milk Dealer *27*, 6, p. 116, March, 1938.

A description of how sweet acidophilus milk can be made in your own plant with no added equipment and at a marked reduction in cost to the consumer. C.J.B.

305. Uniformity of Cream Line. JOSEPH BURNS, Capitol Dairy, Madison, Wis. Milk Dealer *27*, 6, p. 120, March, 1938.

A brief discussion of the importance from a sales standpoint of having a uniform cream line.

The author suggests observation of the following points in order to maintain a uniform cream line.

1. Have definite routine production.
2. Never heat milk above 145° (excepting in flash system).
3. Avoid use of live steam in pasteurizing jackets as much as possible.
4. Have sufficient refrigerating capacity so that milk need not be cooled by water in pasteurizer jacket prior to sending over surface cooler.
5. Avoid excess agitation during holding period and in storage tanks.
6. Avoid holding periods over 30 minutes.
7. Have thermometer in water jacket of pasteurizer so that if heating by hot water, the temperature of the heating medium can be available (usually about 160° to 175°). C.J.B.

Other abstracts of interest are numbers 267, 268, 269, 270, and 271.

PHYSIOLOGY

306. The Immature Rat Uterus as an Assay Endpoint for Gonadotropic Substances. C. G. HELLER, HENRY LAUSON AND E. L. SEVRINGHAUS. Dept. of Medicine, Univ. of Wisconsin, Madison. Amer. J. Physiol. *121*, 2, p. 364, Feb., 1938.

Two hundred and twenty-eight immature female rats were used. The minimal dose producing uterine enlargement (as judged by gross inspection) and uterine weight increases were only one-eighth as large as the minimal dose which resulted in ovary weight increase. The curves rise rapidly to a maximum and very slowly recede, so that only a small portion, at the lowest dose levels, is useful for assay purposes. These workers found that the ovary actually decreased in weight at first as the result of gonado-

tropic stimulation while with larger doses it responds both by increased secretion and growth. An addendum to the paper states that on recalculation of the data of Levin and Tyndale a ratio of 3 or 4 to 1 is obtained, as contrasted to their ratio of 8 to 1. D.L.E.

307. Changes in the Water of Tissues Induced by Diets Containing Various Mineral Supplements. E. S. Eppright and A. H. Smith, Lab. of Physiological Chemistry, Yale Univ. School of Medicine, New Haven. Amer. J. Physiol. *121*, 2, p. 379, Feb., 1938.

The electrolyte balance of the diet influences the hydration of the tissues. When sodium chloride constitutes the only mineral supplement to the salt-poor basal diet of rats, the tissues, except skeletal muscles, are more hydrated than normal. Potassium exerts a slightly modifying effect. When the mineral supplement consists mainly of calcium, the general tendency is toward dehydration.

The normal distribution of water in the organism depends to a considerable extent upon the balance of the calcium with its related elements and the alkali metals sodium and potassium. In the absence of calcium and phosphorus, muscle tissue contains more sodium than can be accounted for by that in the extracellular water as calculated from the chloride present. D.L.E.

308. The Nature of Magnesium Tetany. D. M. Greenberg and E. V. Tufts, Division of Biochemistry, Univ. of California Medical School, Berkeley. Amer. J. Physiol. *121*, 2, p. 416, Feb., 1938.

A study has been made on the influence of a number of factors on the incidence, time of onset, and duration of peripheral vasodilation and hyperirritability in magnesium deficient rats. These symptoms were found to be greatly affected by the degree of magnesium deficiency, the starting age of the rats, and the dietary levels of calcium and vitamin G. At levels of less than 1 mgm. of Mg per 100 grams of food all experimental animals reacted within 10 to 14 days and their total life span was from 21 to 30 days with death resulting from a spontaneous convulsion. Probably the explanation of the apparent synergism of the two deficiencies lies in the fact that vitamin G deficiency itself promoted some damage to the nervous system which would tend to increase its reactivity so that the effects of the two deficiencies are additive. The hissing of an air blast appeared to be peculiarly effective in producing convulsions in hyperirritable individuals.

The localization of the lesion concerned with magnesium hyperirritability appears to be in the midbrain or pons as contrasted with thyroid or low calcium tetany, with which, lesions are more likely located in the neuromuscular junction. At least magnesium tetany differs from calcium tetany in that curare does not prevent the onset of convulsive seizures in this condition.

A later paper of this series (*ibid.*, p. 424) describes the effect of renal insufficiency upon the kidneys. D.L.E.

309. The Influence of Calcium and Potassium upon Intestinal Absorption. J. W. GARDNER AND G. E. BURGET, Dept. of Physiology, Univ. of Oregon Medical School. Amer. J. Physiol. *121*, 2, p. 475, Feb., 1938.

Potassium chloride added to a 10 per cent glucose solution in concentrations from 0.03 per cent to 0.15 per cent increases the rate of absorption of the sugar solution from chronic closed intestinal loops in dogs; in similar concentrations $CaCl_2$ decreases the rate of absorption below normal. The increase produced by a 0.1 per cent solution of KCl is approximately equal to the decrease brought about by a similar concentration of $CaCl_2$. The favorable action of KCl reaches a maximum at about 0.08 per cent under these conditions. The retarding action of $CaCl_2$ increases steadily up to 0.15 per cent which was the highest concentration used. Similar results were obtained with rats by a slightly different technique. The authors state "if these results may be explained by alteration of cell permeability by the two electrolytes, our work conforms to the well established effects of K and Ca ions on cell permeability." D.L.E.

310. Enterocrinin, a Hormone Which Excites the Glands of the Small Intestine. E. S. NASSET, Dept. of Vital Economics, Univ. of Rochester, Rochester, New York. Amer. J. Physiol. *121*, 2, p. 481, Feb., 1938.

Enterocrinin is the name given to an intestinal hormone, not previously described, which plays an important rôle in the secretion of succus entericus. This hormone is obtainable from the small and large intestines of several species of animals. The small gut of the dog and the cow have the highest titre of the animals examined.

The secretagogue activity of crude extracts of these organs is not directly related to blood pressure changes because vasodilation-free enterocrinin has been prepared. Enterocrinin does not excite the pancreas; hence, it is separate and distinct from secretin. It augments the secretion of enzymes as well as fluid, a property usually not ascribed to secretin. D.L.E.

311. The Effects of Hypophysectomy and of Anterior Pituitary Extracts in the Disposition of Fed Carbohydrates in Rats. JANE A. RUSSELL, Institute of Experimental Biology, Univ. of California, Berkeley. Amer. J. Physiol. *121*, 3, p. 755, March, 1938.

In the hypophysectomized rats, the proportion of absorbed glucose oxidized and the proportion of total calories obtained from carbohydrate were both much higher than in the normal rats. Decreases in the amount of

glycogen stored were accounted for by the increases in the rate of oxidation of carbohydrate. It was concluded that the anterior pituitary is concerned not only with preservation of body carbohydrate during fasting, but also with the disposition of this substance when it is fed. D.L.E.

312. Further Studies of Intestinal Absorption with the Performance of Osmotic Work. RAYMOND C. INGRAHAM AND MAURICE B. VISSCHER, Dept. of Physiology, Univ. of Illinois, Chicago, and Univ. of Minnesota, Minneapolis. Amer. J. Physiol. *121*, 3, p. 771, March, 1938.

The authors present a unique theory to explain the different rates of absorption of univalent and polyvalent ions from the intestine. In the presence of poly-univalent salts ($MgCl_2$, $CaCl_2$ and others) and poly-polyvalent salts ($MgSO_4$, $CaSO_4$ and others) uni-univalent salts (such as NaCl) are rapidly absorbed from the intestine against blood plasma concentration gradients as much as 28 to 1 and 10 to 1 respectively. A slow absorption of the polyvalent ions occurs at the same time. While univalent anion impoverishment is occurring in the intestine the pH of the gut fluid invariably becomes more acid, around pH 6.1 to 6.3, while with univalent cation impoverishment the gut fluid becomes more alkaline, reaching pH 7.9.

Without exception ammonia production increases in the gut during this univalent impoverishment. The concentration of NH_3-N in the gut fluid increases to as much as two hundred and fifty times its concentration in the blood. The fact that intravenous injection of ammonium salts produces a very large increase in the NH_3-N content of the gut indicates that the ammonia in the intestinal fluid is of metabolic and not of bacterial origin.

The authors suggest that the selective transport of materials against high concentration gradients is a result of the circulation of fluid through differentially permeable membranes. They believe that there is a continuous flow of fluid into and out of the gut during absorption. D.L.E.

313. The Blood Volume of Normal Dogs. JOHN H. GIBSON, 2ND, JOHN L. KEELEY AND MICHEL PIJOAN, Lab. of Surgical Research and Dept. of Medicine, Harvard Medical School. Amer. J. Physiol. *121*, 3, p. 800, March, 1938.

The merits of the various techniques used in determining blood volume are discussed. Original data on plasma, cell and total blood volume, hematocrit and hemoglobin value of venous blood, and blood velocity rate of 50 dogs are presented. In terms of cubic centimeters per kilogram in dogs of from 5 to 30 kgm. in weight, plasma volume ranged from 41.2 cc. to 51.7 cc.; cell volume, from 36.4 cc. to 54.6 cc.; and total blood volume, from 84.0 cc. to 97.3 cc. No distinct difference in plasma, cell or total blood volume in relation to weight exists between male and female dogs. With increase in body

weight, and the accompanying increase in total blood volume, there is an increase in hematocrit and hemoglobin values, and a slowing of blood velocity rate. This indicates that total blood volume bears a direct relationship chiefly to the amount of muscular tissue in the animal. D.L.E.

314. Further Evidence for a Mammogenic Hormone in the Anterior Pituitary. E. T. Gomez and C. W. Turner, Dept. of Dairy Husbandry, Missouri Agr. Exp. Sta. Proc. Soc. Exp. Biol. Med. *37*, 4, p. 607, Jan., 1938.

Further evidence is presented indicating that the pituitary gland is the seat of production of a factor or factors which stimulate the growth of the duct and lobule-alveolar systems of the mammary gland. This principle, called the "mammogenic hormone," is present in the pituitaries of cattle when pregnant. The growth of the mammary glands of castrated female rabbits and rats was stimulated by the daily injection of such pituitaries. That this principle is not identical with the lactogenic hormone is indicated by the lack of response with non-pregnant cattle pituitaries containing considerable amounts of the lactogenic hormone. S.W.M.

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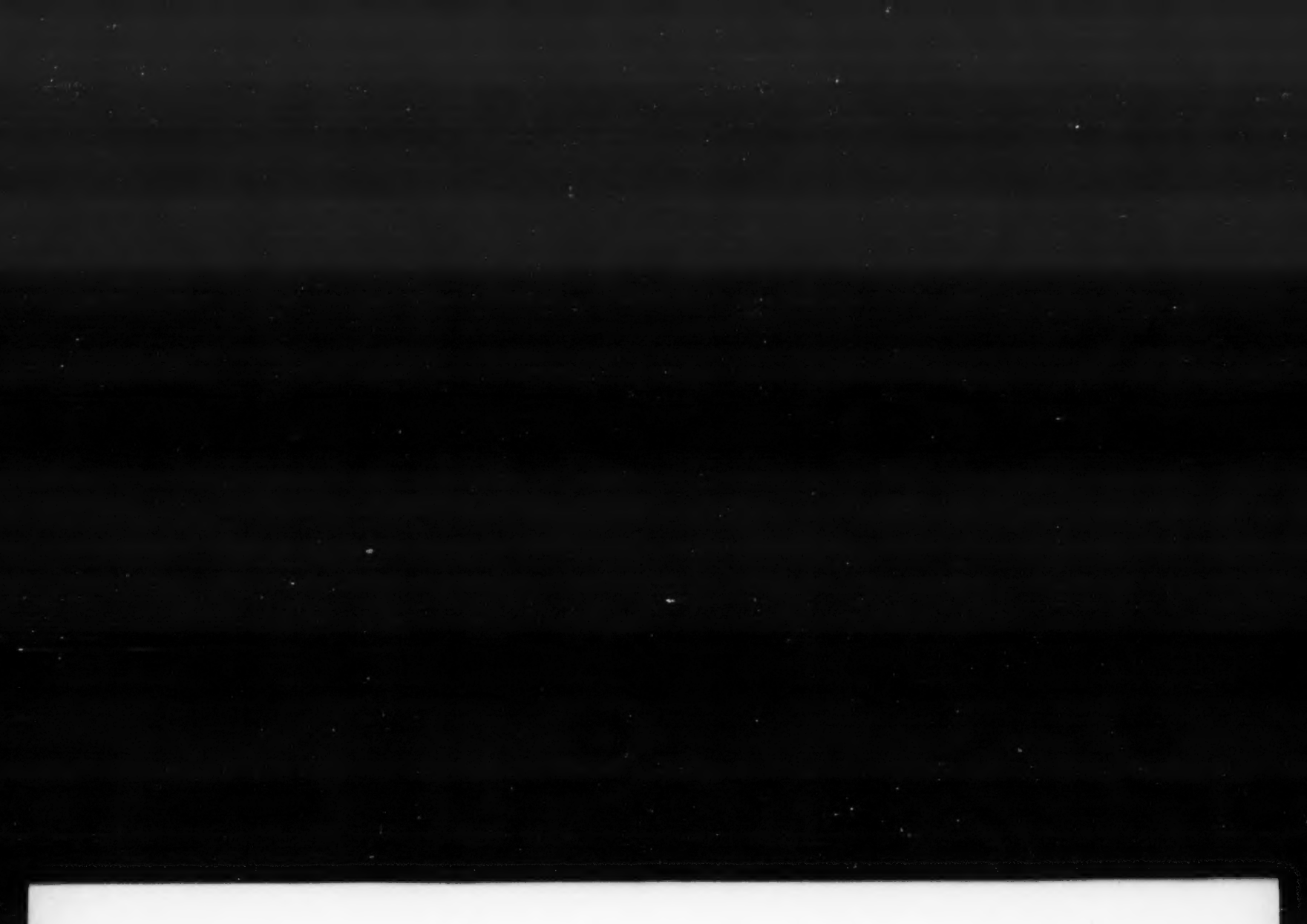

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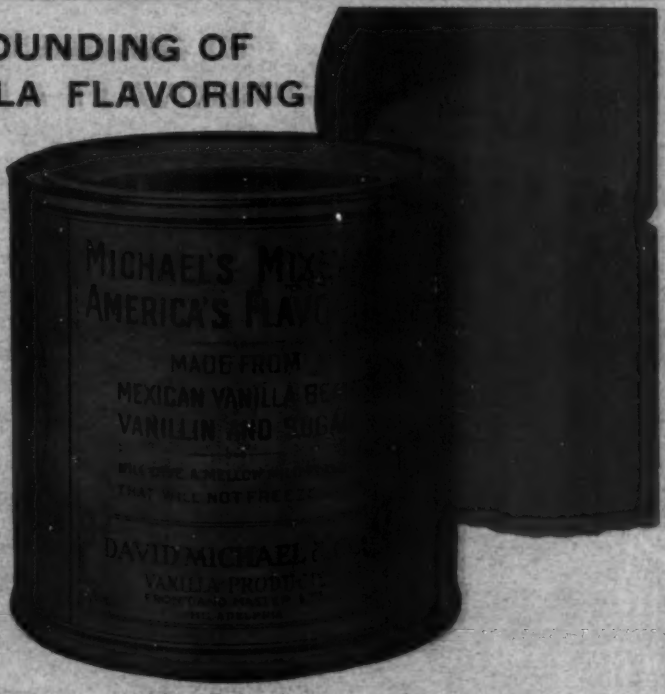

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No producer of milk or dairy products is willing to take chances with flavor. Your sales depends on it. And flavor itself depend on two things—the milk itself and absolute cleanliness of your equipment and your plant.

When you use Wyandotte Cleaner and Cleanser on your equipment, you are *sure* that there will be no "off-flavor" complaints. And, because we can furnish Wyandotte products that are adaptable to any water supply, you'll find that perfect cleaning is easier and more economical.

Let a Wyandotte Service Representative make a survey of your cleaning problems. He will show you the advantages of Wyandotte Cleaner & Cleanser, as well as Wyandotte Detergent for brightening up, and Steri-Chlor, the safe, convenient germicide—all with no cost or obligation on your part.

DIFCO

CULTIVATION OF L. ACIDOPHILUS

Bacto-Trypsin Digest Agar is a most excellent culture medium for propagation of *Lactobacillus acidophilus*. This medium is most widely used in estimating the degree of intestinal implantation and in determining the number of viable organisms in acidophilus products. It is also employed for isolating strains and for carrying stock cultures of the organisms.

Bacto-Trypsin Digest Agar is prepared for use by dissolving the powder in distilled water and sterilizing the solution. It yields uniform lots of media upon which comparable results are secured. The formula used for this medium is that suggested by Dr. H. A. Cheplin.

Specify "Difco"

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In the Research and Development of Bacto-Peptone and Dehydrated Culture Media

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